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Decontamination of Drinking Water Distribution System Infrastructure after Contamination with Untreated Source Water



Office of Research and Development Homeland Security Research Program

# Disclaimer

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Gregory Sayles, Director Center for Environmental Solutions and Emergency Response

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# Abbreviations and Acronyms

atm	atmosphere(s)
AWWA	American Water Works Association
BWS	bulk water sample
CCC	continuing calibration check
CFU	colony forming unit(s)
cm	centimeter(s)
DC	District of Columbia
DPD	N,N-diethyl-p-phenylenediamine
E. coli	Escherichia coli
EPA	Environmental Protection Agency
ft	foot/feet
gpm	gallon(s) per minute
h	hour(s)
HOCl	hypochlorous acid
HSRP	Homeland Security Research Program
in <sup>2</sup>	square inch
INL	Idaho National Laboratory
L	liter(s)
m	meter(s)
mg	milligram(s)
min	minute(s)
mL	milliliter(s)
MPN	most probable number
NTU	nephelometric turbidity units
PEX	cross-lined polyethylene
PPD	presidential policy directive
ppm	parts per million
psi	pound(s) per square inch
PVC	polyvinyl chloride
QC	quality control
QCS	quality control sample
TOC	total organic carbon
WSTB	Water Security Test Bed

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# **Executive Summary**

The U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program partnered with the Idaho National Laboratory (INL) to build the Water Security Test Bed (WSTB) at the INL test site outside Idaho Falls, Idaho. The WSTB was built using an 8-inch diameter cement-mortar-lined ductile iron drinking water distribution pipe that had previously been taken out of service. The pipe was exhumed from the INL grounds and oriented in the shape of a small (450 feet long) drinking water distribution system. The WSTB can support drinking water distribution system research on a variety of topics, including biofilms, water quality, sensors, and homeland security-related contaminants. Since the WSTB is constructed of real drinking water distribution system pipes, research can be conducted under conditions that are representative of the conditions in a drinking water distribution system (USEPA, 2016; USEPA, 2018).

This report summarizes the results of infrastructure decontamination experiments performed at the WSTB. These experiments focused on simulating contamination of drinking water distribution pipes with untreated source water due to a treatment failure at a water treatment facility. A loss of water treatment capability due to an emergency (e.g., power loss) could force a utility to pump untreated source water into the distribution system to maintain basic sanitation (such as toilet flushing), fire protection, and to maintain pressure in the pipes. Compared to treated drinking water, untreated source water will likely have higher levels of various water quality parameters such as turbidity, conductivity, pH and organic carbon, and include increased levels of microbial contamination such as coliform bacteria (including *Escherichia coli* (*E. coli*)). Should an event like this occur, results from this study can help water utilities understand the effectiveness of common distribution system decontamination methods such as flushing and chlorination and decrease the time their systems are offline.

To assess infrastructure decontamination, common processes used by water utilities to clean their pipes were implemented. First, a fire hydrant attached to the 450-ft WSTB distribution pipe was opened, and the water was flushed at approximately150 gpm (0.96 ft/sec) for 20 minutes (min). The pipe was then chlorinated at approximately 55 mg/L, with a contact time of 24 hours (h). After chlorination, the water was flushed through the same hydrant, and uncontaminated local chlorinated tap water was allowed to flow through the pipes. Bulk water samples and samples of the pipe interior were taken during each phase of the experiment to determine if contamination was removed from the distribution pipe.

In addition, a premise plumbing system, which included a hot water heater, refrigerator, dishwasher and washing machine, was contaminated with the untreated water. The system was flushed by opening taps or running the appliance repeatedly. When the 8-inch diameter distribution pipe was chlorinated, this chlorine was allowed to flow into the plumbing and appliances. Preliminary research was also performed with sections of heavily tuberculated unlined cast iron pipe obtained from a drinking water utility. These iron pipe sections were contaminated and decontaminated by filling and draining them, with stagnant water sitting inside the pipe during each phase of the experiment. The goal was to collect preliminary data with heavily tuberculated iron pipe, which may be harder to decontaminate than smoother cement-lined ductile iron pipe.

These experiments were designed to provide water utility operators with realistic expectations of how effective standard decontamination processes like flushing and chlorination would be for returning the distribution system to service. The following is a summary of the results:

- After contamination of the 450-ft distribution pipe, an increase in coliforms/*E. coli* was observed, as well as increases in Total Organic Carbon (TOC), conductivity, pH and turbidity. After flushing water through the fire hydrant at approximately 150 gallons per minute (gpm), no coliforms/*E. coli* were observed in the bulk water or on the pipe surface. TOC, conductivity and turbidity returned to near-baseline levels (pH was more variable). Baseline levels were maintained during chlorination and after tap water was returned to the pipe.
- Contamination from the WSTB distribution pipe entered the premise plumbing system with appliances, which produced an increase in coliforms/*E. coli*, TOC, conductivity, pH and turbidity. Flushing the plumbing pipes, emptying the water tank and running the appliances for a cycle reduced coliforms/*E. coli* to undetectable levels and returned turbidity and conductivity to baseline. TOC also returned to baseline after flushing except for the dishwasher, where increased levels persisted after flushing.
- Individual sections of heavily tuberculated iron pipe were filled with contaminated water, emptied and rinsed (to simulate flushing), then chlorinated and rinsed again. After rinsing (flushing), coliforms/*E. coli* were detected on the inner surface of the corroded iron pipe and in the bulk water. None were detected after chlorination, but coliforms were detected in the bulk water after the chlorine was flushed out.

In summary, flushing uncontaminated tap water for 20 min at 150 gpm (0.96 ft/sec) was effective at removing coliform bacteria from the 450 ft cement-mortar-lined pipe. Flushing was also effective for home plumbing and appliances. In general, water quality parameters such as TOC, conductivity and turbidity returned to pre-contamination baseline levels, suggesting that monitoring water quality might be an effective method of monitoring the progress of decontamination. Coliforms were present after flushing in sections of heavily tuberculated iron pipe, and they appeared in the bulk water after chlorination and a second round of flushing. These data suggest that heavily tuberculated iron may be more difficult to decontaminate via flushing and chlorination than the cement-mortar-lined iron used in other experiments. Also, water quality parameters such as TOC, conductivity and turbidity returned to pre-contamination baseline levels in most cases, suggesting that monitoring water quality might be an effective method of determining the progress of decontamination.

# 1.0 Introduction

## 1.1 Background

The U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program (HSRP) has partnered with the Idaho National Laboratory (INL) to build the Water Security Test Bed (WSTB) at the INL near Idaho Falls, Idaho. The centerpiece of the WSTB is an 8-inch diameter cement-mortar-lined ductile iron drinking water pipe that had been taken out of service. The pipe was exhumed from the INL grounds and then oriented in the shape of a small drinking water distribution system. The WSTB has been fitted with service connections, a premise plumbing and appliance system, fire hydrants, and removable coupons (excised sample materials) to collect samples from the pipe inner surface (USEPA, 2016; Szabo et al, 2017; USEPA, 2018).

Previously, experiments focused on decontamination of various contaminants that adhered to the inner surface of the 8-inch water pipe have been conducted at the WSTB (USEPA, 2016; Szabo et al, 2017; USEPA, 2018). In response to a contamination event, drinking water utilities will likely flush the distribution system using fire hydrants and possibly chlorinate as described in American Water Works Association (AWWA) Standard C-651-05: Disinfecting of Water Mains (AWWA, 2005). The experiments described in this report examine different aspects of distribution system decontamination using these methods.

These experiments focused on simulating contamination of drinking water distribution pipes with untreated source water due to a treatment failure at a water treatment facility. A loss treatment capability due to an emergency (e.g., power loss) could lead a utility to consider pumping untreated source water into the distribution system to maintain basic sanitation (such as toilet flushing), fire protection, and to maintain pressure in the pipes. Compared to treated drinking water, untreated source water will likely have higher levels of various water quality parameters such as turbidity, conductivity, and organic carbon and include increased levels of microbial contamination such as coliform bacteria (including *Escherichia coli* [*E. coli*]). These experiments examine the ability of a water utility to decontaminate water distribution pipes contaminated with untreated source water using flushing and chlorination. This work was extended to premise plumbing, which includes common appliances and water pipes found in homes. The experiments were designed to provide full scale data to utility operators considering using flushing and chlorination to return a water distribution system to service after contamination with untreated source water.

# 1.2 WSTB System Description

A primary feature of the WSTB is an 8-inch (20-cm) diameter cement-mortar-lined ductile iron drinking water pipe oriented in the shape of a small drinking water distribution system. The WSTB contains ports for service connections and a 15-foot (ft) (5-meter [m]) removable coupon section designed to sample the pipe interior to examine the results from contamination/decontamination experiments on the pipe wall. Figure 1 schematically depicts the main features of the WSTB, and Figure 2 shows an overhead view with major components labeled.



Figure 1: Schematic overview of the Water Security Test Bed (WSTB).



Figure 2: Aerial view of the Water Security Test Bed (WSTB).

Drinking water supplied to the WSTB is chlorinated ground water that also supplies the surrounding INL facilities. The WSTB incorporates approximately 450 ft (137 m) of 8-inch (in) (20-centimeter [cm]) diameter cement-mortar-lined ductile iron pipe. The 8-in (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 was estimated to be approximately 1,150 gallons (4,353 liters [L]). The effluent water from the WSTB system was discharged to a lined lagoon (Figure 3) that has a total water storage capacity of 28,000 gallons (105,980 L).



Figure 3: Water Security Test Bed discharge lagoon.

Three individual short sections of pipe were set up next to the lagoon. The individual pipe setup is shown in Figure 4. One pipe section was the same cement-mortar-lined iron pipe used in the 450-ft WSTB pipe. The other two sections were unlined cast iron pipe sections with heavy corrosion (tuberculation) on the interior. Each pipe was approximately 10 ft long. The unlined iron pipes were obtained from the District of Columbia Water and Sewer Authority (DC Water). All pipe surface samples taken from the individual pipes were direct scrapings of the inner surface. Further details on the contamination, flushing, chlorination, and sampling processes are described in Section 2.



Figure 4: Individual pipe sections next to the Water Security Test Bed lagoon (left) and the

#### same pipe sections with the open drainage valves shown (right).

The WSTB also includes a premise plumbing system that is connected to the 8-in diameter distribution main via a 1-in copper service connection (Figure 2). Water flows through the service connection into a water meter, copper plumbing pipes, a removable pipe coupon section with copper, polyvinyl chloride (PVC) and cross-linked polyethylene (PEX) pipes, and appliances including a water heater, dishwasher, washing machine and refrigerator with water dispenser. Water empties into a utility sink with hot- and cold-water taps and then drains to an exterior tank. All water from the tank eventually flows to the lagoon. These components are shown in Figure 5.



Figure 5: Premise plumbing setup at the WSTB: the water meter outside the building (top left), premise plumbing pipes (top middle), water heater (top right), dishwasher, washing machine and refrigerator (bottom left), utility sink (bottom middle) and exterior tank (bottom right).

# 2.0 Description of Untreated Water Contamination and Decontamination Experiments

The following section provides more detailed descriptions of the pipe materials used, how untreated source water was prepared, the procedures used for forming biofilms in the pipes,

contamination and decontamination.

## 2.1 Pipe Materials Tested

Most of the distribution system-sized pipe used at the WSTB for decontamination experiments is constructed of 8-in diameter cement-mortar-lined ductile iron, which was previously excavated from the INL property. This piping had been used as water distribution piping for over 30 years prior to excavation and reuse on this project. Visual inspection of the cement-mortar lining indicated that the lining was in good condition. The interior and exterior view of this pipe is shown in Figure 6.



Figure 6: External and internal view of the cement-mortar lined pipe.

Pipe in the premise plumbing system is mostly 1-in diameter copper, with coupon sections made of copper, PVC and PEX (Figure 5, top middle). The other piping used in this experiment was heavily corroded and tuberculated 8-in diameter unlined iron pipe sections obtained from DC Water. Individual sections of pipe (approximately 10 ft long) were set up next to the lagoon. The individual pipe setup is shown in Figure 4, and internal and external pictures of the pipe are shown in Figure 7. In Figure 4, two of the pipe sections are unlined iron, and one section is cement-mortar-lined iron pipe.



Figure 7: External (left) and internal (right) view of the cast iron pipe with heavy corrosion (tuberculation) on the interior.

# 2.2 Source Waters Used

Two different simulated source waters were evaluated during this experiment. The primary simulated source water used to contaminate the 450-ft distribution pipe, the premise plumbing, and one of the short sections of corroded unlined iron pipe was referred to as "lagoon water". This water was local tap water (chlorinated groundwater) that had traveled through the WSTB pipes and emptied into the lagoon. In addition to this water, the lagoon contains dirt and organic matter such as grasses that have blown in from the surrounding desert. Algae were also present and non-pathogenic *E. coli* K-12 (cultured off site, see Section 2.4) was added to the lagoon. To mix the contents of the lagoon, an individual walked around the lagoon in waders (see Figure 8). Agitation of the water and sediment in the lagoon mixed the *E. coli* and created turbidity in the water. Target water quality in the lagoon was based on the 2018 Annual Report of Water Analysis for the Washington Aqueduct produced by the Army Corps of Engineers (https://www.nab.usace.army.mil/Missions/Washington-Aqueduct/Water-Quality/, last accessed April 7, 2020). In particular, the targets for key water quality parameters were as follows:

- Turbidity: 20 Nephelometric Turbidity units (NTU) (range: 8-33 NTU)
- Total Organic Carbon (TOC): 3.5 parts per million (ppm) (range: 1.2-2.2 ppm)
- Coliforms: 45,000 most probable number (MPN)/100 milliliters (mL) (range: 5,335 to 97,250 MPN/100 mL)

The other water used in the short sections of pipe was collected from the Potomac River downstream from the intake to the DC Water Dalecarlia treatment plant, and shipped by DC Water to the INL WSTB site in a carboy (Figure 8). This source water was not spiked with *E. coli*. This water was used to contaminate two of the individual pipe sections set up next to the lagoon (one corroded iron and one cement-mortar-lined iron). The contamination procedure is further described in Section 2.3.3.



Figure 8: Mixing *E. coli* into lagoon water (left) and a carboy of Potomac River water (right).

## 2.3 Contamination and Decontamination Procedures

Contamination and decontamination took place in the 450-ft distribution pipe, home plumbing and short individual sections of pipe. The following sections describe how contamination and decontamination took place.

# 2.3.1 Distribution System Pipe (450 ft)

Before contamination, biofilm formation was accomplished by passing INL tap water through the pipe continuously for four weeks. After initial flushing to remove any debris, the 450-ft WSTB pipe was set to 2.5 gallons per minute (gpm) for the four-week biofilm formation period. This procedure has been used to form biofilms in the pipe during previous experiments. Forming biofilms using this procedure resulted in biofilm levels of  $10^4$  to  $10^5$  colony forming units (CFU)/square centimeter (cm<sup>2</sup>) in past experiments (USEPA, 2016; USEPA, 2018). These biofilm levels have been consistent between past experiments, and since the same biofilm formation process was used in this study, biofilm levels were not measured.

Contamination of the pipe occurred by pumping the lagoon water (described in Section 2.2) from the lagoon into the upstream end of the pipe, letting it flow through the 450 ft of pipe and emptying back into the lagoon. Pumping lagoon water in a loop kept the pipe full of contaminated water and kept the sediment and *E. coli* in the lagoon well mixed. Water was recirculated in this manner for 18 h (hours). Figure 9 shows the setup used to contaminate the pipe. A hose connected to a swimming pool pump was put into the lagoon (top left) and run the length of the WSTB pipe (top right). On the upstream end, the fire hose that fed tap water to the pipe was disconnected (bottom left), and the line that supplied the lagoon water was attached (bottom right). The pipe was then filled with lagoon water and flow commenced. The pump supplied approximately 10 pounds per square inch (psi) of pressure in the pipe.





Figure 9: The setup used to contaminate the WSTB pipe with lagoon water: hose connected to pump in lagoon (top left), hose from lagoon across WSTB site (top right), disconnection of fire hose (bottom left), and connection of lagoon hose (bottom left).

After the 18-hour contamination period, the first decontamination step was to flush the system through the downstream fire hydrant. Before the flushing began, the pool pump that supplied lagoon water to the 8-inch line was disconnected and the fire hose that normally supplies tap water to the 450 ft pipe was reattached. Once the fire hose with tap water was reconnected, the downstream fire hydrant was used to flush the pipe.

Flushing is shown in Figure 10. A fire hose was attached to the downstream fire hydrant (Figure 10, left) with the outlet placed in the lagoon and braced with sandbags and cement blocks (Figure 10, right). The hydrant was opened as much as possible while keeping the fire hose stable and against the ground. Flow through the hose was estimated to be approximately 150 gpm (0.96 ft/sec). Flushing in this manner took place for 20 min.



Figure 10: Flushing contaminated water from the WSTB distribution pipe via the

# downstream fire hydrant. The left image is the fire hydrant with the hose connected, and the is the hose in the lagoon with sandbags is on the right.

After hydrant flushing was complete, the pipe was chlorinated. Chlorination took place by pumping store-bought concentrated bleach (8.25% hypochlorous acid [HOCl]) into the WSTB pipe. One gallon of bleach was diluted into five gallons of tap water in a carboy and then pumped into the WSTB pipe over a period of one hour with flow through the pipe at 15 gpm. This procedure effectively filled the pipe with chlorinated water. Figure 11 shows a past example of how the disinfectant was added to the pipe.

Once chlorine was detected at the downstream fire hydrant, the flow through the pipe was shut off, resulting in a chlorine concentration of 55 milligrams (mg)/L in the pipe. The water then sat stagnant overnight for approximately 20 h. After the 20-h contact period, the fire hydrant was opened again and flushed for 20 min at a rate of 150 gpm. After flushing, flow through the pipe with tap water (i.e., return to service) was re-established at 2.5 gpm for the duration of the experiment.



Figure 11: Example of how chlorine was introduced to WSTB distribution pipe.

# 2.3.2 Premise Plumbing System

Before contamination, tap water continuously flowed through the premise plumbing system for four weeks to allow biofilm and deposits to form on the inner surfaces of the pipes and appliances. During this pre-contamination phase, total flow though the plumbing system was set to 138 gallons per day (0.096 gpm or 363 mL/min), which is the typical usage in many households (DeOreo et al., 2016). Water flowed through the utility sink taps, with equal flows through the hot and cold water taps. Flow was regulated by a set of flowmeters downstream from the utility sink (Figure 12). The dishwasher and washing machine were operated for one cycle once per week, and the refrigerator water dispenser was opened for 10 minutes once per week.



Figure 12: Flow meters downstream from the utility sink used to control flow through the premise plumbing system.

Contamination of the premise plumbing system was accomplished by allowing lagoon water to flow through the 1-in copper service connection during contamination of the 450-ft distribution pipe. The utility sink's cold tap was opened to allow lagoon water to flow through the plumbing. Once lagoon water reached the cold tap (determined by a spike in turbidity), the hot water tank was emptied through the drain valve at the bottom of the tank, and then refilled with lagoon water. After the hot water tank was filled with lagoon water, the utility sink hot water tap was opened, and the dishwasher and washing machine were run for one cycle. The refrigerator water dispenser was also opened during the contamination phase, but the inline filter quickly became clogged with the particles from the lagoon water. A replacement filter was not available, so the refrigerator water was not sampled during the remainder of the experiment.

Decontamination was performed based on the findings of the Water Research Foundation report 4572 titled "Flushing Guidance for Premise Plumbing and Service Lines to Avoid or Address a Drinking Water Advisory" (WRF, 2016). In this report, suggestions for how to flush household plumbing and appliances were derived from an expert panel of water industry professionals. The specific suggestions summarized in the report are as follows (reproduced verbatim from the report):

#### Flushing Cold Water Taps

- Begin by running the cold-water faucet closest to where water enters the house. Starting from the point closest to where water enters the house, open all the other cold water taps and allow the water to run for 20 minutes.
- Next, flush toilets at least once. If a bathtub has a spout and showerhead, direct flow through the spout.
- Flush all outside spigots for 10 minutes.
- After flushing all cold taps, direct the flow from the bathtub spout to the showerhead, if applicable.

#### Flushing Hot Water Taps and Water Heater

- Run the hot water tap closest to the hot water heater and proceed to open all hot water taps.
- If a bathtub has a spout and shower head, direct flow through the shower head first.
- Allow the water to run for at least 75 minutes and then turn off the faucets.
- If applicable, direct shower head flow to bathtub tap for 2 minutes.

#### Flushing Appliances

- Run empty dishwasher and washing machine once on rinse cycle.
- Replace all water filters (e.g., whole-house filter, refrigerator filter, etc.) and empty ice from ice maker bin; run ice maker and discard 2 additional batches of ice.

Based on the suggestions given in the report, the premise plumbing system was flushed in stages. First, the utility sink hot water tap was closed, the valve to the hot water heater was turned off, and the cold water tap on the utility sink was fully opened. Simultaneously, the cold-water dispenser on the refrigerator was supposed to be opened to its maximum setting. However, as noted earlier, clogging of the refrigerator water system prevented the cold-water dispenser on the refrigerator from being used. Therefore, only the utility sink tap was flushed for 20 min. After the cold-water pipes were flushed, the hot water heater tank was drained and filled with tap water, and the hot water tap in the utility sink was fully opened for 75 min. At the conclusion of the hot water flushing, the dishwasher and washing machine were operated for one cycle.

When chlorine was introduced to the 450-ft WSTB pipe, it flowed into the plumbing system. The hot water tank was emptied and refilled with highly chlorinated water, and elevated chlorine level was confirmed to be present at the hot and cold utility sink taps. Subsequently, the appliances were run for one cycle, and then the whole premise plumbing system sat stagnant for approximately 20 h. After the 20 h stagnation period, the plumbing system was flushed as described above, and then baseline flow was restored for the duration of the experiment (138 gallons per day, or 0.096 gpm (363 mL/min)).

## 2.3.3 Individual Pipe Sections

To compare data on decontamination of heavily tuberculated (corroded) unlined pipe to the much smoother cement-lined pipe from INL, individual sections of pipe were set up next to the lagoon. The setup is shown in Figure 4. In this setup, two pipe sections obtained from DC Water were made of unlined iron with heavy corrosion, and one section was a piece of cement-mortar-lined iron INL pipe. The goal of this experiment was to have decontamination data on both types of pipe contaminated with lagoon water and both pipe types contaminated with Potomac River water. Potomac River water was added to a short (10 ft) section of corroded iron and a separate short section of cement-mortar-lined INL pipe. Lagoon water was used to contaminate the other corroded section (the middle pipe section in Figure 4) of DC Water pipe. A cement lined pipe section from INL was not contaminated with the lagoon water, since data was already available for the prior experiment with the 450 ft of INL pipe.

Pipe sections were set up on a rack as shown in Figure 4. It was not practical to form biofilms in the short pipes in the same way as the 450-ft distribution pipe or home plumbing system. Therefore, before contamination, the inner pipe surfaces were wetted with local tap water from a

garden hose. Surfaces were completely wetted over the course of approximately three minutes. After wetting, the pipe sections were filled with either lagoon or Potomac River water. Potomac water shipped from DC Water was poured directly into a DC Water and an INL pipe, and a separate carboy was filled with lagoon water using and poured into a DC water pipe. Water was poured into the pipe sections directly from the carboy or using a bucket. As seen in Figure 4, the pipe sections had removable caps on each end that kept the pipes full and were used for drainage. Each pipe also had a valve that was used to allow air to escape and reduce pressure on the pipe caps. Once contaminated, the pipe sections sat stagnant for 20 h.

After contamination, the pipe caps were removed, and the pipes were drained. Flushing was simulated by running tap water over the inner pipe surfaces for five minutes using a garden hose. Chlorination was accomplished by filling the pipes with chlorinated water and allowing the pipes to sit stagnant for another 20 hours. Chlorine was made using store-bought concentrated bleach, and concentrations in the pipe sections ranged from 60 to 150 mg/L. The range in values is likely due to variation in chlorine demand in different pipes, which is difficult to predict. After the chlorination period, the pipes were emptied and rinsed with a garden hose. The pipes were then filled with tap water and allowed to sit for one day before being emptied and rinsed. Bulk water and scrape samples were collected from the pipe sections at each step in the process.

## 2.4 Experimental Methods

Methods used to conduct the decontamination methods at the WSTB site are described in this section. Non-standard methods are described in detail. Standard methods or methods with publicly available references are noted by referencing the method but are not described in detail.

#### Preparation and transport of E. coli stock

An *E. coli* suspension was produced by mixing an inoculum of *E. coli* cells in nutrient broth and incubating at 37 °C for 24 h. The resulting stock concentration was  $1 \times 10^{11}$  CFU/100 mL. The resulting prepared stock was shipped to the INL site in two separate 500-mL containers inside coolers at  $4 \pm 2$  °C. This suspension was dumped into the lagoon waters and mixed by having an individual walk around in the lagoon in waders and mix the water.

#### Extraction of biofilm and adhered E. coli from coupon and pipe surfaces

Removable cement-mortar coupons in the WSTB pipe were sampled by shutting off water flow to a small section of the pipe containing the coupons, draining water to relieve pressure, and unscrewing the coupons. The coupons were scraped with a sterile scalpel (Thermo Scientific, Waltham, MA) into a sterile sample bottle (Thermo Scientific, Waltham, MA) containing sterile buffer (Sigma Aldrich, St. Louis, MO) while periodically rinsing the scalpel with sterile buffer.

Pipe surface samples were taken directly from the inner surface of the tuberculated pipe sections. The biofilm, corrosion and spores were scraped from the surface using a disposable sterile surgical scalpel (Thermo Scientific, Waltham, MA). An O-ring (Grainger, Lake Forest, IL) with an area of 0.371 square inches (in<sup>2</sup>) (2.4 cm<sup>2</sup>) was placed on the pipe wall, and the area inside the O-ring was scraped to ensure that the same area was scraped for each sample.

For each type of sample, the extracted material was collected in a sterile sample bottle with a sodium thiosulfate tablet (Thermo Scientific, Waltham, MA) (for dechlorination of the water)

and 100 mL of pre-filled carbon-filtered water. The extracted sample was transferred to a cooler at  $4 \pm 2$  °C. The samples were shipped cooled overnight to the EPA laboratory and analyzed upon receipt but within 24 h of sampling. As noted in the results, some samples were analyzed in the field for coliform presence/absence.

#### Bulk Water Sampling

The Bulk Water Sample (BWS) for coliforms/*E. coli* and other water quality parameters were collected using the grab sampling technique in 100 mL sterile sample bottles with a sodium thiosulfate tablet. The bulk water sampling port in the WSTB coupon section was opened and the water was drained for 15 seconds prior to collection of 100 mL of water from the WSTB.

#### Laboratory and Field Enumeration of Coliforms/E. coli

Upon receipt in the laboratory, coliform and *E. coli* samples were immediately analyzed using Colilert-18 (IDEXX Corp, Westbrook, ME), which conforms to method 9222D in Standard Methods for the Examination of Water and Wastewater (APHA, 2005). Samples analyzed in the laboratory were quantified for the number of coliforms/*E. coli*. Samples analyzed in the field were noted for presence or absence of coliforms/*E. coli*.

#### Total Organic Carbon

Upon receipt in the lab, TOC samples were analyzed via EPA Method 9060a (https://www.epa.gov/hw-sw846/sw-846-test-method-9060a-total-organic-carbon, last accessed April 7, 2020). Samples were preserved via addition of acid and had a holding time of 28 days, so analysis was not always immediate upon receipt.

#### Free Chlorine

Free chlorine samples were analyzed immediately in the field using the Hach Method 10102 using N,N-diethyl-*p*-phenylenediamine (DPD) (<u>https://www.hach.com/asset-get.download-en.jsa?code=55578, last accessed April 7, 2020</u>). Samples were diluted in distilled water as needed.

#### Turbidity, Conductivity, pH and Temperature

These parameters were analyzed immediately in the field. Turbidity was measured using a Hach 2100P Portable Turbidimeter (Hach Corp., Loveland, CO), and all analyses and calibration followed the manufacturer's instructions. Conductivity, pH and temperature were measured by a YSI 556 multiprobe sonde (Xylem, Rye Brook, NY). All analyses and calibrations followed the manufacturer's instructions.

## 2.5 Quality Control and Data Quality

## 2.5.1 Quality Control

Quality control (QC) samples for the contaminant reference method included continuing duplicate samples, controls and laboratory blanks. The data quality objectives for each of these quality control samples are provided in Table 1. The acceptable ranges limit the error introduced into the experimental work. All analytical methods operated within the QC requirements for controls and laboratory blanks, and unless otherwise noted in the Deviations (Section 2.5.3), all data quality objectives in Table 1 were met. Note that duplicate samples for *E. coli* refer to a

duplicate analysis of one sample. All *E. coli*, free chlorine and TOC samples were collected in duplicate.

Measurement	QA/QC Check	Frequency	Acceptance Criteria	Corrective Action
Total Coliform/ <i>E. coli</i>	Positive Control	Every batch of samples	Positive – Total coliforms – all wells yellow <i>E. coli</i> – all wells fluorescing	Use new media vessel and dilution buffer
	Negative Control		Negative – Total coliforms – No yellow wells <i>E. coli</i> – No fluorescent wells	
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.
Turbidity	Check standard set for 2100 P	Once per day	Deviation of ± 0.2 NTU	If it fails, repeat calibration
Conductivity, temperature, pH	Calibration	Once per experiment	As specified by manufacturer	If it fails, repeat calibration
Total Organic Carbon	Calibration Curve (5-point minimum)	When new standards are made (30-day hold time) or a CC** has failed	r-value ≥ 0.993	Prepare fresh standards and analyze again
	Calibration Blank	One per batch of 20 field samples	[blank] ≤ 0.35 mg/L Organic Carbon	Suggest carryover or contamination. Troubleshoot method or instrument and correct.
	Quality Control Sample (QCS); also called Initial Calibration Check	Immediately after calibration	80% to 120% recovery	Remake standard and if that fails, recalibrate with fresh calibration standards
	Continuing Calibration Checks (CCCs)	After every 10 <sup>th</sup> field sample and at the end of the sequence	Vary concentrations for longer sequences (low to mid to high) Low $\pm$ 50% Mid $\pm$ 20% High $\pm$ 15%	Instrument response may have drifted. Troubleshoot and recalibrate if needed. All field samples should be bracketed by acceptable Continuing Calibration Verifications (CCVs)
	Duplicate samples (Field duplicates when possible, Laboratory duplicates if not)	One pair per batch of 20 field samples	Relative Percent Difference (RPD) < 20%	If field duplicates fail, run laboratory duplicates. Failing field duplicates could be sample collection or matrix issues if the laboratory duplicates pass.

Table 1: Quality control data quality objectives

Measurement	QA/QC Check	Frequency	Acceptance Criteria	<b>Corrective Action</b>
				If laboratory duplicates fail, instrument troubleshooting is needed
	Matrix Spike	One per batch of 20 field samples	Recover 70% - 130% of spiked concentration when compared to a duplicate sample	Rerun – continued failure suggests matrix interference

\*DPD = N,N-diethyl-phenylenediamine sulfate \*\*CC=calibration check

#### 2.5.2 Data Quality

At least 10% of the data acquired during the evaluation were audited. These data include the biofilm *E. coli* measurements, BWS and water quality measurements. The data were traced from the initial acquisition, through analysis, to final reporting, to ensure the integrity of the reported results. All calculations performed on the data undergoing the audit were checked. No significant adverse findings were noted in this audit.

## 2.5.3 Deviations

When conducting scrape samples from the interior of the drinking water pipes, the sampling method calls for using an O-ring to isolate the area to be sampled. Scraping within the O-ring area was meant to standardize the pipe surface area that was sampled. The tip of a scalpel was to trace the sampled area inside the O-ring. It was observed in the field that the traced area was not always an exact circle. Therefore, the area sampled may have varied between samples. In some cases where tuberculation was heavy, the O-ring was not used at all, and the scraped area was estimated. It was not possible to precisely quantify this variation. It should also be noted that the level of tuberculation varied between pipes, and spatially within individual pipes. However, it was estimated that the sampled area could have varied by 5% between samples and this variation should be considered when interpreting the data.

Another deviation was observed with the Colilert-18 *E. coli* presence/absence testing in the field. The interaction of the *E. coli*-spiked lagoon water with the heavily corroded DC Water short pipe section and increased levels of chlorine during that phase of the experiment created a yellow tinted water. When coliforms are present, the Colilert-18 test turns water samples yellow. The water samples from this pipe were already yellow before they were placed in the incubator. The sample did appear to turn a darker shade of yellow overnight, indicating the possible presence of *E. coli*, but this result was a judgment call. The over-chlorination step for the DC short pipe section (filled with lagoon water) was repeated and the yellow tint was much lower but still yellow prior to incubation. The laboratory sample results should be used to provide the final determination of coliform presence for this pipe section.

## **3.0 Experimental Results**

#### 3.1 Decontamination of the 450-ft 8-in Diameter Distribution Pipe

Figure 13 and the figures that follow in this section show the various phases of the

decontamination experiment. Background is the baseline level in the water or on the pipe surface before contamination. The contamination phase is when lagoon water was introduced into the pipe. The flushing phase is when the downstream fire hydrant was opened for 20 min at a flow of approximately 150 gpm. Chlorination is when chlorine was introduced to the pipe, and the water in the pipe sat stagnant for roughly 20 h. Return to service occurred after the chlorine was flushed from the pipe, and local tap water flow was reintroduced.

Figure 13 shows that coliforms/*E. coli* were not detected in the tap water flowing through the pipe before contamination, but they increased upon introduction of the lagoon water. After flushing the pipe for 20 min, the coliforms/*E. coli* levels in the bulk water retuned to non-detectable. The same was true after chlorination and during the return to service phase. The data suggest that flushing alone may remove coliforms/*E. coli* from the cement-mortar lined distribution pipe. However, only one data point after flushing exists, which was followed by chlorination. Flushing followed by chlorination should be sufficient to remove coliforms/*E. coli* in untreated river water from cement-mortar lined iron pipe.



Figure 13: Contamination and decontamination of coliforms/*E. coli* in the bulk water phase of the 450 ft cement-mortar lined iron distribution pipe.

Figure 14 shows that the number of coliforms/*E. coli* adhered to the interior surface of the cement-mortar lined iron pipe followed the same trend as in the bulk water phase. The number of adhered coliforms/*E. coli* spiked during contamination but returned to non-detectable levels after flushing. No detectable coliforms/*E. coli* were observed after chlorination or return to service. The data suggest that flushing alone may remove coliforms/*E. coli* from the lined distribution pipe interior surfaces. However, only one data point after flushing exists, which was followed by chlorination. Flushing followed by chlorination should be sufficient to remove coliforms/*E. coli* in untreated river water from the distribution pipe interior surfaces.



Figure 14: Contamination and decontamination of coliforms/*E. coli* from the pipe surface of the 450-ft cement-mortar-lined iron distribution pipe.

Figure 15 and Figure 16 show how water quality parameters changed during each phase of the decontamination experiment. Distinct spikes in conductivity, turbidity and TOC were observed during the contamination phase, with decreases following in the flushing, chlorination and return to service phases. In the decontamination phases, turbidity and TOC returned to baseline levels. After the contamination phase, conductivity was 4% lower than the baseline levels. However, it is unclear if the decrease relative to baseline was a true decrease in the conductivity of the water or drift in the conductivity sensor. Compared to the baseline levels, a drop in pH was observed during contamination. However, in the decontamination phases, pH levels were variable and below the baseline. The data suggest that TOC, turbidity and possibly conductivity could be parameters that could indicate that untreated water has been removed from the distribution system.



Figure 15: Conductivity and turbidity measurements during each stage of the

#### decontamination experiment.



Figure 16: TOC and pH during each stage of the decontamination experiment.

# 3.2 Decontamination of the Premise Plumbing System

The figures in this section show the results from the background, contamination and decontamination sampling in the home plumbing system. Background is baseline level of each parameter coming out of the water taps or water in the appliances before contamination. The contamination phase is when lagoon water was introduced into the pipes and appliances. The flushing phase is when the taps were flushed, the hot water heater drained and refilled, and the appliances run for one cycle (without detergent). Chlorination is when chlorine was introduced to the pipes and appliances and allowed to sit for approximately 20 h. Return to service occurred after the chlorine was flushed from the pipe, and local tap water flow was reintroduced into the plumbing and appliances.

Figure 17 and Figure 18 show the coliform and *E. coli* levels in the plumbing and appliances during the phases of the experiment. In both figures, no microbial contamination was detected in the baseline phase, which was expected. Increases in coliform and *E. coli* were observed in all pipes and appliances. After following the flushing procedure described in Section 2.3.2, no coliforms or *E. coli* were detected in the hot- or cold-water pipes, or in any of the appliances. The same result was found after chlorination. The data suggest that flushing the hot- and cold-water pipes according to the procedure described in Section 2.3.2 removed coliform and *E. coli* contamination that came from untreated water.



Figure 17: Total coliform levels in the bulk water phase of the pipes and appliances in the home plumbing system.



Figure 18: *E. coli* levels in the bulk water phase of the pipes and appliances in the home plumbing system.

Figure 19, Figure 20, Figure 21 and Figure 22 show the change in TOC, pH, turbidity and conductivity, respectively, during the phases of the contamination and decontamination experiment. In Figure 19, TOC increased during the contamination phase relative to the baseline. In the hot and cold taps and the hot-water heater, TOC levels returned to baseline levels after flushing and running the appliances and remained at that level after chlorination. In the washing machine, TOC levels returned to baseline after chlorination, but levels remained elevated after the flushing phase. The reason for this result is unclear, but it is possible that some untreated water was trapped in the washing machine after one cycle. It is possible that the washing machine may need to be run for multiple cycles before all untreated water can be

cleared.

TOC levels in the dishwasher were elevated relative to the pipes and other appliances in the baseline phase and remained elevated after flushing and chlorination. This elevation of TOC levels has been observed in previous decontamination studies conducted with the home plumbing setup (Szabo et al., 2017). This result is likely due to organic compounds leaching from the plastic components of the dishwasher. Still, TOC values could indicate when untreated water has been removed from plumbing pipes, the hot-water heater, and possibly the washing machine. If the dishwasher is primarily plastic on the inside, TOC values should be used cautiously.



Figure 19: TOC levels in the bulk water phase of the pipes and appliances in the home plumbing system.

Figure 20 shows pH values during each phase of the experiment. The pH value generally decreased as contamination, flushing and chlorination took place. However, it is possible that this decrease is due to natural variation in the pH of the source water, and it is unlikely that this variation could be used to determine when untreated water had been removed from the plumbing and appliances. Conversely, Figure 21 and Figure 22 show that turbidity and conductivity, respectively, increased during contamination. Similar to the 450 ft distribution pipe, turbidity returned to its baseline value after flushing and chlorination, but conductivity was 3-5% lower than baseline. It is unclear if the decrease relative to baseline was a true decrease in the conductivity of the water or drift in the conductivity sensor. The data suggest that turbidity and possibly conductivity could be parameters that could indicate that untreated water has been removed from the plumbing system pipes.



Figure 20: pH levels at the hot and cold taps in the home plumbing system.



Figure 21: Turbidity levels at the hot and cold taps in the home plumbing system.



Figure 22: Conductivity levels at the hot and cold taps in the home plumbing system.

## 3.3 Decontamination of individual corroded iron pipe sections

As described in Section 2.3.3, individual short sections of pipe were set up next to the lagoon. Two of these sections were made of cast iron and had a heavily corroded (tuberculated) inner surface, and one section was cement-mortar lined water pipe. Lagoon water was added to one section of corroded iron pipe, and water from the Potomac River was added to one corroded iron and one cement-mortar pipe section. This allowed for data collection with lagoon water on both types of pipe (one short corroded iron section, and the 450-ft cement-mortar-lined pipe) and real source water from the Potomac River on both types of pipe sections.

TOC and other water quality parameters were not monitored in the short pipe sections. Data collection focused exclusively on coliform analyses. Figure 23 shows the number of coliforms in the bulk water phase in the individual pipe sections during each phase of the experiment. Potomac River water coliform levels were between 10-20 MPN/100 mL inside both types of pipes. After the river water was drained, and the pipe section was "flushed" with a garden hose, no coliforms were detected in the bulk water when the pipe was refilled. No coliforms were detected after chlorination in either pipe. The same trend was observed with coliforms adhered to the inner pipe surface (Figure 24).

Figure 23 also shows the level of coliforms in the bulk water phase in a corroded iron pipe section filled with lagoon water. Lagoon water was substantially more concentrated in coliforms than Potomac River water, with approximately 10<sup>4</sup> MPN/100 mL. After draining the pipe section filled with lagoon water, flushing with a garden hose and then refilling, coliforms were still observed in the bulk phase. Figure 24 shows that coliforms were also detected on the pipe surface after flushing. No coliforms were seen in either phase after chlorination. However, once chlorination was complete, and the pipe was refilled, coliforms were observed in the bulk water phase (2 MPN/100 mL). These data suggest that coliform remained adhered to the corroded iron pipe surface after chlorination but detached from the pipe surface and were detected in the water.

The results from these experiments indicate that that coliforms in the range of 10-20 MPN/mL from untreated Potomac River water can be removed from the corroded iron (DC Water) and cement-mortar lined (INL) iron pipe with "flushing" and chlorination (possibly with only flushing). However, the data from these pipe sections is a small-scale representation of an actual distribution pipe, and the results should be replicated on a larger scale. For the cement-mortar-lined pipe, lagoon water with three orders of magnitude more coliforms were successfully decontaminated from the 450-ft distribution pipe with flushing and chlorination. Therefore, it is likely that coliforms from Potomac River water could also be successfully decontaminated using these techniques on the full scale.

Compared to lagoon water results in the 450-ft pipe cement-mortar pipe, coliforms were more persistent on the corroded iron pipe section (Figure 23). It is possible that multiple rounds of flushing and chlorination would be needed to remove them. However, for the lagoon water tests, the corroded iron (pipe section) and cement-mortar lined pipe (450-ft distribution pipe) experimental setups were not the same, and caution should be used when comparing the results. In the future, adding corroded iron pipe sections to the 450-ft distribution pipe and repeating these experiments with untreated lagoon water would shed light on whether the results can be replicated at a larger scale.



Figure 23: Total coliform levels in the bulk water phase of the individual short pipe sections.



Figure 24: Total coliform levels on the inner pipe wall of the individual short pipe sections.

# 4.0 Conclusions

The following points summarize the results of the untreated water contamination and decontamination experiments performed at the WSTB:

- After contamination of the 450-ft distribution pipe, an increase in coliforms/*E. coli* was observed, as well as increases in TOC, conductivity, pH and turbidity. After flushing water through the fire hydrant at approximately 150 gpm, no coliforms/*E. coli* were observed in the bulk water or on the inner pipe surface. The same was true after chlorination and returning tap water flow to the pipe. However, only one sampling event took place after flushing alone is adequate to remove microbial contamination from the cement-mortar lined pipe. If possible, the impact of above ground storage tanks, distribution network complexity (e.g. pipe loops and other configurations) and various flow velocities will be investigated.
- In the 450-ft distribution pipe, TOC, conductivity and turbidity returned to near baseline levels after decontamination (pH was variable). Baseline levels were maintained during chlorination and after tap water was returned to the pipe. The data suggest that TOC, turbidity and possibly conductivity measurements could indicate that untreated water has been removed from the distribution system. Using ultraviolet-visible light-based on-line TOC sensors in real time to detect changes in TOC is also a topic of interest.
- Increases in coliforms/*E. coli* TOC, conductivity, pH and turbidity were observed in the plumbing pipes and appliances after contamination. Flushing the plumbing pipes, emptying the hot water tank and running the appliances for a cycle reduced coliforms/*E. coli* to undetectable levels, and returned turbidity and conductivity to baseline. Like the 450-ft distribution pipe, it might be informative to repeat these experiments using flushing only.

- In the plumbing system, TOC returned to baseline after flushing and chlorination in the hot- and cold-water pipes and the hot-water heater. A spike in TOC was observed in the washing machine after the flushing cycle, possibly due to untreated water being trapped in the appliance. TOC returned to baseline after the chlorination step. TOC data from the dishwasher were elevated during all phases, likely due to organic material leaching from the plastic surfaces. The data suggest that TOC (in the situations noted above) could indicate that untreated water has been removed from the plumbing system. Turbidity and possibly conductivity might also be parameters that could indicate that untreated water has been removed from the plumbing pipes.
- Individual sections of heavily tuberculated iron and cement-mortar-lined iron pipe were filled with contaminated water, emptied and rinsed (to simulate flushing), then chlorinated and flushed. Potomac River water with coliform/*E. coli* levels in the 10-20 MPN/100 mL was successfully flushed from the pipe with no microbial contamination remaining in the water or on the pipe surface.
- After flushing, coliforms/*E. coli* from untreated lagoon water were detected on the inner surface of the corroded iron pipe and in the bulk water. None were detected on the pipe surfaces after chlorination, but coliforms were detected in the bulk water after the chlorine was flushed out. This differs from the decontamination results using untreated lagoon water in the 450-ft cement-mortar-lined pipe. However, the experimental systems used for the pipe types were different, and it would be informative to repeat these experiments on the full scale with corroded iron pipe built into the 450-ft distribution pipe system.

In summary, flushing and chlorination were effective at removing coliform bacteria from cement-mortar-lined infrastructure and home plumbing and appliances. However, it would be informative to repeat the contamination experiments in the 450-ft distribution pipe and home plumbing to confirm that flushing alone is an effective decontamination technique without chlorination. Water quality parameters such as TOC, conductivity and turbidity returned to precontamination baseline levels in most cases, suggesting that monitoring water quality might be an effective method of determining the progress of decontamination. This information would be useful to utility responders since parameters like TOC, conductivity, and turbidity are easier and faster to obtain with field instruments than *E. coli*, which requires lengthy incubation times (18 to 24 h). Coliforms were present after simulated flushing in one section of heavily tuberculated iron pipe, and they reappeared in the bulk water after flushing and chlorination. These data suggest that heavily tuberculated iron may be more difficult to decontaminate than the cement-mortar lined iron used in other experiments. It would be informative to repeat these experiments at full scale with corroded iron pipe built into the 450-ft distribution pipe.

#### 5.0 References

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