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Standard Operating Procedures: Sorbent Onsite Testing Apparatus (SOTA)

NESDI Project #578
Task 4

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ADMINISTRATIVE INFORMATION

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CONTENTS

FIGURES

TABLES

1. INTRODUCTION

Per- and polyfluorinated alkylated substances (PFAS) are a diverse group of chemicals that are widespread in groundwater systems including at US Navy and US Marine Corps sites and may present human and ecological health risks. PFAS are currently removed from groundwater by pumping the water out of the ground through various wells, passing the water through large containers of sorbent material (i.e., granulated activated carbon (GAC)), and then pumping the treated water back into the ground (Figure 1). There is some evidence that different types of sorbent material (for example, anion exchange resin) may be differentially effective at treating PFAS-impacted groundwater, and that different background water quality conditions (i.e., salts, organic carbon) may also impact the efficacy of these sorbent-based treatments. Therefore, testing different types of sorbent material at a given site to determine the likely efficacy based on site conditions can help site managers select an appropriate sorbent material for a given site.

The procedures in this document describe the general process to deploy and use the Sorbent Onsite Testing Apparatus (SOTA, Figure 2) designed for NESDI project #578 to assess the efficacy of different types of sorbent materials to treat PFAS-impacted water at a field site using either a rapid small scale column testing and/or a full-scale column testing approach. Some details of the general process may vary depending on the given conditions at a particular site and selection of numbers and types of columns used. The specific choices used for on-site testing at former Marine Corps Air Station Tustin for NESDI project #578 are also described to help illuminate the methods described in this document. While this device was developed for use with PFAS sorbents, it could be used to test the efficacy of different sorbents to treat other contaminants in natural waters, provided that appropriate tubing and container materials are used for a particular contaminant to reduce the possibility of artifacts.

Figure 1: Part of the South pump-and-treat system installed at former Marine Corps Air Base Tustin.

Figure 2: Overview photo of the SOTA (Sorbent Onsite Testing Apparatus) in use at former Marine Corps Air Base Tustin.

2. MATERIALS

The following lists indicate materials required to replicate the SOTA deployment conducted for NESDI Project #578. Quantities required will vary depending on the length of a given deployment, the frequency of sampling, and the number of different columns used for a given project. Guidelines to calculate these quantities are provided in italics text below.

General laboratory materials:

- □ HDPE 250 ml sample bottles *Quantity: Sufficient bottles for all samples at all timepoints, plus at extra set of bottles equal to the number of columns tested. Additional bottles should also be purchased to allow for replacements in the case of a spilled/contaminated sample.*
- □ 1 quart zip-top bags *Quantity: Sufficient bags should be purchased to allow all samples to be double-bagged. Additional bags should also be purchased to be used for storing sample tubing, ice, and other needs that may come up.*
- □ 1 gallon zip-top bags *Quantity: Sufficient bags to use ~10-12 bags for ice and chain of custody paperwork for each sample shipment, plus extras.*
- □ Powder-Free Nitrile Gloves *Quantity: 2 boxes of gloves of the correct size per person are recommended*
- □ Pre-washed cotton lab coats – *1 for each person of the correct size*
- □ Squirt bottle for methanol
- □ Safety glasses – *1 for each person of the correct size*

- □ Ear plugs – *1 set for each person for each day expected to be working at field site*
- □ pH meter
- □ Conductivity meter
- □ Field balance
- □ Sharpie® and ballpoint pens
- □ Scissors
- □ Colored laboratory tape and packing tape
- □ Kimwipes™
- □ Large absorbent laboratory wipes
- □ Large plastic bins for storing supplies for field effort – *Quantity: 3-4 bins should be sufficient, depending on size and quantities of materials.*
- □ Coolers for shipping samples to analytical laboratory (standard 48 quart size) – *Quantity: Approximately one cooler per 40-50 samples. Following Draft EPA method 1633 for analysis of PFAS in natural waters, samples must be stored in a refrigerator, and the analytical lab must extract them within one month of the date the samples were taken.*
- □ Carboy of deionized (DI) water
- □ Beaker
- □ Bucket
- □ (Large diameter) tubing of sufficient length and correct diameter to refill water drums with site test water, if needed

Chemicals and sorbents:

- □ Sorbent 1 – can be ground or unground, depending on specific experimental needs
- □ Sorbent 2 – ground (180-212 µm) and unground
- □ (Sorbent 3, etc. if desired – ground (212-180 µm) and unground)
- □ Optima Methanol (MeOH)

SOTA and column materials:

- □ SOTA, consisting of:
    - Pelican case housing overall SOTA
    - Multi-channel Peristaltic pump or diaphragm pumps on shelf
    - Column holders
    - GFCI extension cord
    - Female HDPE and stainless steel quick-connect bulkhead adapters with 1/8” hose barbs (8, installed for inlet treatment water)
    - Female HDPE and stainless steel quick-connect bulkhead adapters with 1/4” hose barbs (8, installed for outlet waste water)
- □ Glass econo-column chromatography columns. *Diameter of columns used depends on particle size of tested sorbent; the sorbent particle size: column diameter ratio should be 1:50. In the example setup used here, the ground sorbent had an average particle size of 200 µm, so the minimum column diameter was 200x50 µm = 1,000 µm, or 1 cm in diameter. For the unground sorbent with average particle size 750µm, the minimum column size required was 3.75 cm; 5 cm diameter columns were selected for unground sorbent testing based on available sizes. Purchase sufficient quantity of selected column sizes to accommodate desired tests, with some additional backups in case of breakage or other problems.*

- □ Glass wool – *1 package*
- □ Glass beads (diameter should be similar to the tested sorbent particle size to ensure the sorbent bed is held in place. In the example test shown here, 0.5 mm diameter beads were used) *Quantity: Calculate the maximum amount needed based on the number and size of columns to be used. Typically, the glass beads should be placed on both the top and bottom of the sorbent, which should be approximately centered vertically within the column.*
- □ Zip ties – various sizes. *Recommend purchasing small colored zip ties to help visually differentiate influent, effluent, sample collection, and possibly passive sampler tubing, etc., For example, purchasing at least 25 small cable ties of each color for each column used would provide sufficient ties and allow for extras. Approach described below used 56 blue (inlet water), 32 red (waste water), 24 yellow (sample water) and 8 gray (water flowing to passive sampler) zip ties.*
- □ Tubing – make sure to select tubing sizes that will best meet experiment needs. Typically, if using peristaltic pumps, larger diameter tubing and slow flow speeds will extend the life of the tubing. For PFAS, silicone tubing is recommended. It is best for each column to have an individual line and pump driving the water, and to have the tube diameter the same or to increase in diameter as it moves through SOTA, if needed (not decrease in size). This will limit air bubble and backpressure issues through the system. See below for our example setup:
    - HDPE hose barb splice (straight) adapters 1/8" ID *Quantity: minimum 16; used to connect pump tubing to other sections of inlet tubing*
    - HDPE male luer lock to hose barb adapters 1/8" ID *Quantity: minimum 8; used to connect inlet water lines to stopcocks at bottom of test columns; add at least 2 more if one inlet tubing line will be plumbed to accommodate a passive sampler*
    - HDPE female luer lock to hose barb adapters 1/4" ID *Quantity: minimum 12; used to connect passive sampler bypass lines and small column sampling lines*
    - HDPE and stainless steel female quick-connect adapters with 1/4" hose barbs *Quantity: minimum 8; used to provide easy sample collection access at end of sample tubes*
    - HDPE male luer lock to hose barb adapters 1/4" ID *Quantity: minimum 12; used to connect passive sampler inlet and waste lines and large column sampling lines*
    - HDPE male quick connect to hose barb adapters 1/8" ID *Quantity: minimum 8; used to connect inlet water lines pulling from drums to bulkhead quick connect adapters installed in field apparatus system*
    - HDPE male quick connect to hose barb adapters 1/4" ID *Quantity: minimum 16; used to connect outlet waste water lines to bulkhead quick connect adapters installed in field apparatus system to deliver waste water to collection vessel, and to deliver sample water into sample bottles*
    - 3-way luer lock stopcocks with one female and two male ends *Quantity: minimum 24; used for tops and bottoms of all test columns and passive sampler holders; add at least 2 more if one inlet tubing line will be plumbed to accommodate a passive sampler*
    - Female to male luer lock swivel adapters *Quantity: minimum 12; used to connect inlet water line to bottom 3-way stopcocks for all test columns and to connect top of passive sampler holder to top 3-way stopcock; add at least one more if one inlet tubing line will be plumbed to accommodate a passive sampler*
- □ Silicone tubing, platinum cured, 1/8"
- □ Silicone tubing, platinum cured, 1/4"

- □ Sampling rack(s): container such as a clear plastic bin that holds 8-10x 250-ml bottles upright (1 or 2)
- □ Sampling lids: 250-ml bottle lids, drilled with two holes: one to accommodate 1/8" peristaltic tubing, and a second smaller hole for air venting – *Quantity equal to the number of columns to be used*
- □ Inlet water source: *Depending on site setup, several design options are possible. First, if the groundwater well, or source of water for the study, can accommodate the study flow speed, water can be pulled directly from the system into the SOTA. In cases where the system flow speed is too high, or low, for the SOTA setup, a secondary container may be required to temporarily hold test water that is then pulled through the SOTA system. A secondary container also avoids potential issues with variations in flow from the site water system. In the example study described here, the site groundwater pumping system had significantly higher flow rates than the SOTA could accommodate, so HDPE water drums (55-gallon drums) were used to store test water. The example test flow rate required drums to be refilled every week during the deployment. Note that for this second method, the persistence of contaminants and other water quality parameters should be considered when selecting a hold/refill time, to avoid unrealistic changes to water quality parameters as much as possible.*
- □ Passive samplers, if desired (for example, diffusive gradient in thin film [DGT] samplers) *Quantity: calculate the number of samplers needed for desired number of columns and number of timepoints to be sampled. For example, the test conducted for this project connected DGT passive sampler devices to four sample columns and one inlet water line (5 total sampler devices) and intended to replace samplers at 4 timepoints, requiring a total of 20 DGTs)*

3. CLEANING PROCEDURES

To reduce contamination of samples from PFAS in the environment, the following procedures should be followed. These should be modified if using the SOTA for other contaminants.

PFAS-clean best practices should be followed; this includes avoiding the use of PFAS-containing clothing, food, and other products before sampling and/or at the sampling site. A clean cotton laboratory coat and clean nitrile gloves should be worn during all sampling activities and during handling of components of the field apparatus.

The following cleaning procedures should be used for field apparatus deployment and testing areas, as possible:

1. Wipe down work surfaces (i.e. work table) with soapy water for initial cleaning of dirt; air dry
2. Wipe down work surfaces with methanol prior to use
3. Regularly control dust deposition by wiping down as above

Cleaning procedures for sample bottles and reusable equipment:

1. Rinse with MeOH at least 3x
2. Air dry in a clean environment using a plastic shield to prevent deposition of particles

3. Place clean bottles and equipment inside clean plastic bags for storage and transport

Cleaning of materials between tests/samples that will come into contact with test water/samples:

1. Rinse 3x with MeOH
2. Rinse 2-3x with DI water

4. SOTA TEST PREPARATION

4.1 PLAN TESTS

1. Identify the desired sorbents or combinations of sorbents to test for a given site.
2. Ideally, each sorbent tested (and particle size, whether full scale or RSSCT), will be run in duplicate. Each test should also include at least one column with no sorbent as a positive control. The SOTA has the ability to supply influent water to 8 different columns, which can be either all full scale, all RSSCT, or a combination of full scale and RSSCT columns. Iterate options for the column layout to achieve test goals.
3. Identify the average particle size for the sorbents to be tested, prior to any grinding (from manufacturer).
4. Identify the target empty bed contact time (EBCT) for the sorbents to be tested (from manufacturer).
5. Identify the target ground particle size to be used for RSSCT, if using.
6. Use either the continuous diffusion model and/or proportional diffusion model to calculate the target EBCT for the RSSCT columns for each sorbent (see Crittenden et al. 1991). It has been suggested for PFAS that if the TOC is expected to exceed 2.5 mg/L C in the test water, the proportional diffusion model should be used, unless existing data shows that the continuous diffusion model will work (Schaefer et al. 2020).
7. Calculate the time to reach a target bedvolume for the study (for reference, many studies use 30,000 bedvolumes of water treated; study time = bedvolume*EBCT), for both full scale and RSSCT columns, if using.
8. Adjust the target ground particle size if desired, and recalculate the EBCT for RSSCT and the time to reach a target bedvolume to select the target particle size to achieve study goals, within logistical constraints (i.e. personnel availability, project timelines, etc.). Smaller particle sizes will result in lower EBCT and shorter times to reach target bedvolumes of water treated.
9. Select a sorbent layer thickness, and calculate the volume of the sorbent bed at this thickness for each column.
10. Calculate the flow velocity to achieve the target EBCT based on the sorbent bed volume (flow velocity = sorbent bed volume/EBCT).
11. Calculate the total volume of water to be used for each column to reach the target bedvolume (volume of water needed for each column = study time * column flow rate).
12. Calculate the total volume of water to be used across all columns for the entire study, and over various periods of time (i.e. per day, per week, etc.).

13. Iterate sorbent layer thicknesses to select an appropriate thickness/sorbent volume that will result in an acceptable flow velocity and water volumes used over time, given logistical constraints (i.e. consider flow rates achievable with pumps on hand, speed of drawing down test water, rate of waste water generation, etc.).

4.2 PREPARE MATERIALS FOR FIELD TESTS

1. Prepare test water drum(s): wash test water drum following above procedures, and cover with clean plastic for transport to field test site to prevent dust/external contamination from entering the drum.
2. Prepare sorbents:
    a. For RSSCTs:
        i. Use a blender to grind GAC sorbent(s) with a small amount of water. GAC is too soft to use a dry grinder (it gets pulverized too finely). Work in small batches to avoid excessive waste of material.
        ii. Grind AIX sorbents (dry) in a dry grinder (cleaning the grinder before and after each use). The grinder should be used in a fume hood.
        iii. If using other sorbents, test small batches of material to determine an appropriate grinding method.
    b. Wash (ground and/or unground) sorbents through sieves with DI water to select the correct size fraction.
        i. For example, use a #70 sieve in sequence with a #80 sieve to select the 180-212 µm particle range for RSSCT tests. Work in small batches to avoid excessive waste of material.
        ii. Use a sieve finer than the target sorbent size to wash excess fine materials from sorbents for full scale column tests. Work in small batches to avoid excessive waste of material.
    c. Dry sorbents in HDPE containers at least overnight at 40°C in a drying oven to help evaporate some of the DI water.

4.3 PREPARE COLUMNS

1. For the NESDI project #578 field test, a total of 8 columns with sorbent materials were used: 4 small (RSSCT, using ground sorbent) and 4 large (full scale, using unground sorbent) columns.
2. For each column, first puncture the frit with a clean (methanol-wiped) metal needle. This was determined necessary during laboratory testing to allow sufficient water flow through the columns to meet target empty-bed contact times for each sorbent material; unpunctured frits prevented sufficient water flow speeds. Water was prefiltered with 10-µm in the test system used, such that these frits were unnecessary for filtration.
3. Fit a 3-way stopcock “T” with one female luer lock and two male luer lock ends to the bottom of each column such that the middle male luer lock is connected to the bottom of the column (resulting in the “T” being “upside down”).

4. Each column is then packed as follows with a methanol-slurry-packing method (Figure 3, see details in the RSSCT testing procedures document):
    a. With 3-way stopcock on the column base turned such that flow out the bottom of the column is off, fill the column with methanol; intermittently open the valve to drain methanol as needed while the column is being filled. Also, tap the side of the column to release air trapped between the beads/sorbent granules and pack the beads/granules into place during filling.
    b. Add glass beads to the bottom of the column. For NESDI project #578, a 3.5 cm layer of glass beads was used.
    c. Add a layer of sorbent material of sufficient thickness to result in the target EBCT calculated above. For NESDI project #578, GAC RSSCT columns had 3 cm of sorbent, GAC full scale columns had 1.5 cm of sorbent, and AIX RSSCT and full scale columns had 1 cm of sorbent (ground and unground, respectively), added above the beads.
    d. One RSSCT column should contain no sorbent, and will just be filled with glass beads, as a PFAS blank.
    e. After adding sorbents, add additional glass beads until the column is nearly filled (~0.5cm below the top of the column, to the base of the plastic collar).
    f. Each of the columns is then packed with a small amount of wetted glass wool to hold the column material in place.

Figure 3: Packed columns with GAC and AIX. Columns on left are full scale, smaller columns on right are RSSCT columns.

4.4 PREPARE SOTA

4.4.1 Install columns and tubing

1. Packed columns are placed into the field-testing unit at the lab by pushing each small column into an empty space in the small column holders, or by clamping each large column into an empty space.
2. Tubing is placed in the unit and connected to the columns as follows (described in sequential order from inlet to outlet).
3. Lengths of 1/8” silicone tubing measured to reach the bottom of the test water drums are connected to the hose barbs on male quick-connect to hose barb fittings. Small zip ties (color indicating inlet water; blue in example photos) are placed to secure the tubing to the hose barb, and the quick-connects are then connected to the female bulkheads at the top of the SOTA.
4. Appropriate lengths of 1/8” silicone tubing are placed to connect between the hose barbs inside the unit attached to the female bulkheads and the pump tubing, using a 1/8” barbed splice adapter between this length of tubing and each pump tube. Tubing is secured to barbs with zip ties (color indicating inlet water).
5. Each 3-stop silicone pump tube (2.79 mm inner diameter) is loaded into a cartridge, each of which are then connected onto the appropriate pump. The other end of the pump tube is connected to another 1/8” barbed splice adapter and secured with a zip tie (color indicating inlet water). Each pump tube should be labeled to indicate the column number that it is supplying water to, using a marked wrap of lab tape.
6. Appropriate lengths of 1/8” silicone tubing are connected to the other end of the 1/8” barbed splice adapters attached to each pump tube to connect to 1/8” hose barb-male luer lock adapters that will be connected to supply inlet test water to the female side of the 3-way stopcock at the bottom of each test column. Lengths should be cut generously to provide some extra material to allow for tubing to be shifted as needed within the SOTA during testing. Tubing should be secured to the 1/8” hose barb-male luer lock adapters using zip ties (color indicating inlet water).
7. Connect the inlet tubing to the female side of the 3-way stopcock at the base of each column using a female-male luer lock swivel adapter. This will allow the inlet tubing luer lock to be snugly attached to the stopcock at the base of each column without introducing twists in the tubing which could lead to unscrewing and leaks in the system.
8. For all large columns, a 3-way stopcock “T” with one female luer lock and two male luer lock ends should be connected to the top of each column such that the outer male luer lock is connected to the top of the column (resulting in the “T” being “sideways”; see Figure 4, left). This stopcock can then be turned either to direct water flowing out of the column “sideways”, to flow past the passive sampler and then to waste (for those columns that will be connected to a passive sampler), or to flow directly to waste (for those columns that will not be connected to a passive sampler), or can be turned to direct water vertically into the sampling tubes to collect a sample.

9. For all small columns, a 3-way stopcock “T” with one female luer lock and two male luer lock ends should be connected to the top of each column such that the middle male luer lock is connected to the top of the column (resulting in the “T” being “right side up”; see Figure 4, right). This stopcock can then be turned either to direct water flowing out of the column towards the back of the SOTA, to flow past the passive sampler and then to waste (for those columns that will be connected to a passive sampler), or to flow directly to waste (for those columns that will not be connected to a passive sampler), or can be turned to direct water towards the front of the SOTA, into the sampling tubes to collect a sample.

Figure 4: (Left) Top of one of the full-scale columns that is plumbed with a 3-way stopcock to flow either to a passive sampler and then to waste (right tube, marked with gray and white hashed zip tie), or to sample collection (top tube, marked with yellow zip tie). (Right) Top of one of the RSSCT columns plumbed with a 3-way stopcock to flow either to a passive sampler and then to waste (right tube, marked with gray and white hashed zip tie), or to sample collection (left tube, marked with yellow zip tie).

10. For all large columns, connect lengths of 1/4" silicone tubing to the female side of the 3-way stopcock from the last step using a 1/4" hose barb to male luer lock fitting, secured with a zip tie (color indicating sample water, yellow in example photos; see Figure 2, left). Tubing should be long enough to be secured to the bottom of the pump shelf and extend a short ways out the open front of the SOTA to allow samples to be collected (see Figure 3).
11. For all small columns, connect lengths of 1/4" silicone tubing to the other male side of the 3-way stopcock at the top of the column using a 1/4" hose barb to female luer lock fitting, secured with a zip tie (color indicating sample water, yellow in example photos; see Figure 2, right). Tubing should be long enough to be secured to the bottom of the pump shelf and extend a short ways out the open front of the SOTA to allow samples to be collected (see Figure 3).

12. At the end of the sample tubing, attach a 1/4" hose barb to female quick-connect adapter, and secure with a zip tie (color indicating sample water). Label the end of each of these tubes with the column number using lab tape of the same color as the sample water zip tie indicators, and label each quick-connect adapter with the column number as well, to allow for easy sample identification. Secure these sample tubes to hold them in place below the pump shelf with large zip ties (see Figure 5).

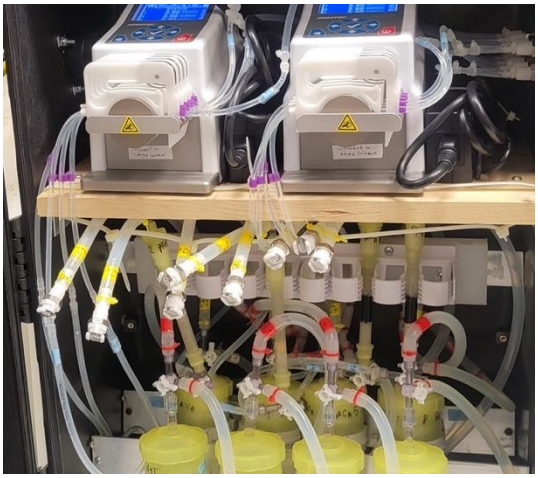

Figure 5: Sample tubes (marked here with yellow zip ties and lab tape) are terminated with female quick-connect adapters and are secured to the bottom of the pump shelf using large zip ties, allowing easy access to collect samples.

13. For all columns that do not flow to a passive sampler, the water that flows out of the top of the column will go directly to waste if it is not being directed to collect a sample. Therefore, connect a length of 1/4" tubing to the open end of the 3-way stopcock at the top of each column using a 1/4" hose barb to female (for large columns) or male (for small columns) luer lock fitting (see Figure 2). Secure the tubing to the hose barb using a zip tie (color to indicate water flowing to waste; red in example photos). The other end of this tubing should be connected to flow out of the SOTA through another set of female bulkhead quick-connect fittings (similar to the inlet bulkhead fittings, but with 1/4" hose barbs) into a waste collection container. Note that in example photos, waste tubing was plumbed together using “Y” hose barb fittings to result in a single outlet line from the SOTA; however, this approach is not recommended and will be adjusted for future applications to provide individual outlet lines for each column. Secure the lines to the outlet hose barbs with zip ties (color indicating waste water)
14. Connect a length of 1/4" tubing to the outside of the female quick-connect bulkhead for waste water using a male quick-connect with a 1/4" hose barb. Secure the tubing to the hose barb with a zip tie (color to indicate waste water). Ensure this tubing is long enough to be secured to the top of the waste collection container to allow water to flow into the container, but short enough to prevent the end of the tubing from becoming submerged in the waste water (which would cause back-pressure in the system).
15. For all columns that flow to a passive sampler before flowing to waste, connect a length of 1/4" tubing to the open end of the 3-way stopcock at the top of each column using a 1/4" hose

barb to female (for large columns) or male (for small columns) luer lock fitting. Secure the tubing to the hose barb using a zip tie (color to indicate water flowing to passive sampler; gray with white hashing in example photos). The other end of this tubing should be connected to a 1/4" hose barb to male luer lock fitting, secured using a zip tie (color to indicate water flowing to passive sampler).

16. Passive samplers are housed within otherwise empty large-scale glass columns. Each of these columns should be fitted with a 3-way stopcock "T" with one female luer lock and two male luer lock ends such that the middle male luer lock is connected to the bottom of the column (resulting in the "T" being "upside down"). The length of tubing from the prior step should be connected to the female side of this 3-way stopcock.
17. The top of each passive sampler column should be fitted with a female-male luer lock swivel adapter, and then with a 3-way stopcock "T" with one female luer lock and two male luer lock ends should be connected to the top of each column such that the outer male luer lock is connected to the top of the swivel adapter (resulting in the "T" being "sideways").
18. Next, a bypass line should be connected between both remaining male ends of the 3-way stopcocks on the top and bottom of each passive sampler column. This will allow water to be directed to flow around the column when passive samplers need to be removed and replaced at intervals during the test. This bypass line should be comprised of 1/4" tubing connected to 1/4" hose barb to female luer lock adapters at both ends, secured with zip ties (color to indicate water flowing to waste). The tubing should be long enough that it does not pull against the luer lock fittings, but short enough that it is not in the way. See Figure 4 for an example of a passive sampler column plumbed with incoming water from a test column and the bypass line described in this step.

Figure 6: Large column holding a diffusive gradient in thin film (DGT) passive sampler plumbed to receive outlet water from column number 1, with a bypass line (marked with red zip ties) to allow water to continue flowing while DGTs are changed out during the test.

19. Finally, the line to deliver waste water from the passive sampler (either from water that has flowed through the column holding the passive sampler, or from the bypass line) should be plumbed by connecting a length of 1/4" tubing to the female end of the top 3-way stopcock using a 1/4" hose barb to male luer lock connector. Secure the tubing to the hose barb using a zip tie (color coded to indicate waste water). The other end of this tubing should be connected to flow out of the SOTA through the outlet female bulkhead quick-connect fittings (similar to the inlet bulkhead fittings, but with 1/4" hose barbs) into a waste collection container.
20. If desired, a passive sampler system can be attached to the inlet water line that will be used to deliver water to the quality control (no sorbent) column using the same approach (with bypass tubing to allow passive sampler exchange) described above, but for 1/8” tubing.

4.4.2 Flush system at laboratory

1. Stopcocks at the bottom and top of each passive sampler container should be oriented to direct water through the bypass tubing and to waste at this time.
2. With all stopcocks at the bottom of columns still in the column-closed position, prime the tubing using DI water and flush out any air bubbles from the system. Once water starts flowing through the tubing, it will drip out of the side of the 3-way stopcock below each column (which is not connected to any tubing). Use towels and/or a containment device placed below the SOTA to collect water. Once all air bubbles are flushed from the tubing, turn stopcocks to allow water into the columns.
3. Once water has begun flowing out of the columns and into the waste capture container, pause pumps and move tubing into methanol.
4. All columns and tubing material will be rinsed with methanol for 20-30 mins at the experimental flow rate as a cleaning step at the lab. Collect and dispose of the methanol waste following site hazardous waste collection and disposal guidelines.
5. After rinsing the packed columns and tubing with methanol, pause pumps and return tubing to DI water, avoiding introducing new air bubbles into the system. Flush out any bubbles if observed. All columns will be flushed with DI water overnight; DI waste can be disposed in the lab sink or following alternative site procedures.
6. If using materials for the first time, it is advisable to collected a sample of DI water after the flushing procedure to ensure that material selections are appropriate (not leaching contaminants, etc.).

4.4.3 Prepare system for transport to field site

1. The following morning, stop the pumps and close the valves at the top of each column; this will prevent water from continuing to drain and/or reverse flow.
2. Remove the inlet and outlet lines for the system from the bulkhead connectors, placing the lines into clean plastic zip-top bags.
3. Unplug the field test unit at the bulkhead on the side of the unit.
4. Gently place clean bubble wrap inside the unit behind, below, and in front of columns to hold columns and tubing in place during transit.

5. Close and latch the lid of the SOTA unit.
6. Wrap unit in clean plastic wrap on all sides, but ensure water can drip out of emergency drain hole if needed.
7. Transfer field unit to test site in upright orientation at all times. A wheeled cart can be used to transfer the unit between the lab and the vehicle that will be used to drive the unit to the test site.

5. SOTA TEST EXECUTION

5.1 DAY 0 (START OF TESTING)

5.1.1 Site set up:

Note that some details described here may require adjustment based on site specific conditions

1. Place SOTA on a sturdy and level work surface. For example, the test conducted at the former Marine Corps Air Station Tustin used a pre-cleaned plastic folding table set up inside a shed that houses that on site pump and treat equipment. The table was placed next to the untreated water tank to allow the SOTA effluent to be easily disposed directly back into the untreated water tank.
2. Wearing gloves, a clean lab coat, and protective eyewear, remove plastic wrap from SOTA, open lid and gently remove bubble wrap. Check for any damage or leaks.
3. Plug SOTA into site power using GFCI cable and extension cord. Secure extension cord to prevent tripping and/or pulling on the unit using zip ties and/or duct tape.
4. Using DI water (or contaminant free water), place the influent lines in the container and pump the water through the system to obtain a negative blank control sample from each line.
5. Place labelled influent drum on casters near the valve from which it will be filled with filtered, untreated site water.
6. Fill influent drum with filtered, untreated site water.
7. Wheel the influent drum closer to the SOTA.
8. Connect influent lines to bulkheads on exterior of SOTA and place the ends of the lines into the influent drum, taking care to keep the lines clean during this process.
9. Cover the top of the influent drum with clean plastic wrap or secure a large zip-top bag over the top of the drum to prevent dust or other material from entering the container. Add an identifying label to the exterior of the drum(s).
10. Connect effluent line(s) to bulkheads on exterior of SOTA and secure the end(s) of the line(s) to allow effluent water to flow into the untreated water tank, ensuring that the ends of the lines will not become submerged.

5.1.2 Start experiment:

1. Turn stopcocks on the base of all columns to close the bottom of the columns, and direct water to the open side of the 3-way stopcock, to allow air to be purged.

2. Turn stopcocks on the top of all columns to open the top of the columns and direct water to waste. Because the bottom stopcocks are closed, water in the effluent lines will not backflow through the columns.
3. Start peristaltic pumps, ensuring any bubbles that may have entered the system during transit and set up are purged, as described above. Again, use disposable towels or a water collection vessel to dry/collect water that drips out of the stopcocks below each column during this air purging step.
4. When air is sufficiently purged from each line, turn the stopcock at the base of each column to direct water through the column.
5. Note down the date and time the water was directed through each column during the prior step, beginning the experiment for that column, in a data log sheet.
6. Orient the 3-way stopcocks at the bottom and top of each passive sampler holder column (which will be dry and empty at this stage) to direct water flowing out of the test columns through the passive sampler holder column instead of the bypass. The previously empty columns will now slowly fill with treated water.

5.1.3 Water quality, procedural blank and sample collection:

1. Set up water quality/sample collection rack: place 8x dry, pre-numbered (according to the column numbers) and pre-weighed (with dry weights recorded and labelled on bottles) HDPE bottles into the rack.
2. Working one at a time, remove a clean, labeled water sample collection lid with a pre-installed sample collection line and male quick-release connector from its labelled bag, and connect it to the correct numbered sample line in the SOTA. Place the attached bottle top and end of the sample line into the labelled bag. Complete for all sample lines.
3. After at least 5 minutes have passed since the experiment started, with site water flowing through columns, one by one, adjust the valves above each column to direct water from the waste line/passive sampler line to the sample collection line for each column.
4. Once water starts dripping into the labelled bags from the end of a sample line, that sample line should be attached to the respective water quality sample bottle by screwing the lid onto the water quality bottle.
5. Record the time water started being collected into each water quality bottle.
6. Allow 20 minutes or so for the sample bottles to fill with enough water to use with the water quality probes and to weigh the water accumulated to confirm flow rates. When sufficiently full, switch the 3-way valves at the top of each column to direct water to waste/passive sampler container instead of the sample lines, noting down the time the water quality sample stopped being collected.
7. Water retained in the sample line should be allowed to flow into the water quality sample bottle by disconnecting the line from the quick-connect attachment to allow air to displace the water in the line. The sample line should then be re-attached to the sample line quick-connector.
8. Working with one sample at a time, unscrew the water quality sample and place aside for weighing and water quality analysis. Then, attach a pre-cleaned sample bottle to the lid

attached to the sample line and place back in the bottle rack. Sample bottle lids should be retained in the clean plastic bag in which clean sample bottles are stored in batches, until needed.

9. When ready, direct water into the sampling tubing again by switching the 3-way stopcock at the top of a given column. Collect a small amount of water into each bottle, swish this water around the inside of the sample bottle, then unscrew the bottle and pour out this water into a temporary waste container (this material will be disposed by adding back into the untreated water tank). Repeat to rinse the inside of the sample bottle with sample water 3x.
10. After rinsing, replace lids with hoses on the sample bottles and leave in place to fill sample bottles (Figure 7), noting collection start time for each sample on data sheet.

Figure 7: SOTA in operation at field site, during sample collection step.

11. As sample bottles fill, carefully weigh each water quality bottle using a portable lab scale and record the weight to the smallest decimal available. This will be used to estimate flow rates. After weights are taken, analyze pH, temperature, and conductivity using handheld meters. When finished, dispose of the liquid in the untreated water tank on site, and rinse each water quality bottle 3x with DI water.

12. Once sample bottles are filled, switch the valves above each column to redirect treated water into the waste/passive sampler effluent lines. Carefully remove the sample collection lids from each sample bottle and immediately cap each bottle with a clean cap.
13. Double bag and label samples using ballpoint pen or Sharpie® on the interior bag.
14. Disconnect the sample lid lines from the SOTA and store each in a dedicated labelled zip-top bag.
15. Note down the time each sample collection was completed.
16. Repeat until all samples have been collected and bagged.
17. Place samples in refrigerator at 4°C (<6°C) until shipment to the analytical facility.

5.1.4 Passive sampler addition, collection and changeout:

1. Once columns intended to hold passive samplers are full and excess water is flowing out through the waste water lines, passive samplers can be added to these containers. To do so, first switch the passive sampler column holder flow to the bypass setting.
2. Next, working one at a time, carefully remove a passive sampler holder column from the SOTA and unscrew the top 3-way stopcock from the swivel adapter.
3. Remove the top of the passive sampler holder column.
4. Remove a passive sampler from its clean bag and place inside the passive sampler holder column. Some water will be displaced, so this step should be completed over a container that will capture the excess water. Orient the passive sampler appropriately according to the manufacturer; for DGTs, the exposure window should be facing upwards in the column. Clean forceps may be required to adjust the orientation of the passive sampler.
5. Note that time to the minute that the passive sampler was placed into the test water.
6. Replace the top to the passive sampler holder column, then screw the 3-way stopcock back into place above the swivel adapter.
7. Place the column back into the SOTA.
8. Redirect water through the passive sampler holder column.
9. To remove and replace passive samplers at timepoints during the experiment, repeat steps 1-3, then remove passive sampler carefully with clean forceps.
10. Once removed, rinse passive sampler with DI water, and place in a small labelled zip-top bag. Note the recovery time to the minute.
11. Repeat steps 4-8 to place a new passive sampler to replace the one that was removed.
12. Store DGTs at 4°C

5.1.5 Influent control collection:

1. Collect approximately 5 ml of influent water into a clean 250 ml HDPE container, swish, and discard into the large untreated water tank; repeat for a total of 3x
2. Fill up the same 250 ml container with influent water.
3. Cap, double bag and label influent sample, with the correct timepoint (Day 0 for the first day of the experiment, etc.).
4. Note down the date and time sample was collected.

5. Place sample in refrigerator until shipment to analytical laboratory for initial concentration analysis.

5.2 SUBSEQUENT TEST DAYS

Following the sample collection timeline determined for the experiment (an example is shown) below, on subsequent test days, collect influent samples and/or effluent samples from indicated columns, and replaced passive samplers as desired, following procedures described above.

Table 1: Sorbent/Timepoint table showing sample collection dates and counts of samples for SOTA test at Tustin.

Timepoint #	1	2	3	4	5	6	7	8	9	10	11	12	Total
Day #	T0	T3	T7	T11	T16	T22	T28	T35	T42	T49	T56	T63	
	Number of samples to collect at each timepoint from each column												
1 AIX full scale A	1	1	1	1	1	1	1	1	1	1	1	1	12
2 PFAS blank	1	1	1	1	1	1	1	1	1	1	1	1	12
3 AIX full scale B	1	1	1	1	1	1	1	1	1	1	1	1	12
4 AIX RSSCT	1	1	1	1	1	1	1	1	1	1	1	1	12
5 GAC full scale A	1	1	1	1	1	1	1	1	1	1	1	1	12
6 GAC RSSCT A	1	1	1	1	1	1	1	1	1	1	1	1	12

Timepoint #	1	2	3	4	5	6	7	8	9	10	11	12	Total
Day #	T0	T3	T7	T11	T16	T22	T28	T35	T42	T49	T56	T63	
7 GAC RSSCT B	1	1	1	1	1	1	1	1	1	1	1	1	12
8 GAC full scale B	1	1	1	1	1	1	1	1	1	1	1	1	12
Influent site water	1	1	1	1	1	1	1	1	1	1	1	1	12
DGTs													
DGT influent water					1		1		1			1	4
DGT (columns 1,4,5,7)					4		4		4			4	16
DGT Blanks	5												

Timepoint #	1	2	3	4	5	6	7	8	9	10	11	12	Total
Day #	T0	T3	T7	T11	T16	T22	T28	T35	T42	T49	T56	T63	
Ship 1			**X**										*27 (5 DGTS)*
Ship 2						**X**							*27 (5 DGTs)*
Ship 3								**X**					*18 (5 DGTs)*
Ship 4										**X**			*18 (5 DGTs)*
Ship 5												**X**	*18 (5 DGTs)*
Total samples													108 (25 DGTs)

5.3 SHIPPING SAMPLES

At various points in the experiment, samples must be shipped to the analytical laboratory. When shipping samples, complete a chain of custody (CoC) form, and place all samples and blanks in one or two shipping coolers with wet ice separately double bagged. CoC should be included in one of the coolers, inside of a dry zip-top bag. Tape the cooler(s) shut with packing tape, and ship overnight to the analytical laboratory.

Please see details below.

5.3.1 Sample Handling and Storage Requirements

1. Samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection.
2. Sample temperature must be confirmed to be at or below 6°C when the samples are received at the laboratory.
3. Samples stored in the lab must be held at or below 6°C until extraction, but must not be frozen.
4. Samples must be delivered on wet ice to the analytical laboratory with sufficient lead time to allow the laboratory to extract the water samples within 28 days of laboratory testing.
5. Samples should be shipped double bagged with wet ice in separate bags to avoid cross-contamination and should be labeled using sharpie on the interior bag.

6. REFERENCES

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14. ABSTRACT

This is the standard operating procedure (SOP) for version 1 of the sorbent onsite testing apparatus (SOTA), as it was deployed in NESDI project 578

15. SUBJECT TERMS

Per and polyfluoroalkyl substances; perfluoroalkyl acids; granulated activated carbon; anion exchange resin; sorption; remediation

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