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Advanced Oxidation Assisted Groundwater Treatment System: A Field Evaluation

Office of Research and Development

Center For Environmental Solutions and Emergency Response

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ADVANCED OXIDATION ASSISTED GROUNDWATER TREATMENT SYSTEM: A FIELD EVALUATION

ORD Project Lead: Vasudevan Namboodiri, Ph.D., ORD/CESER/Homeland Security and Materials Management Division (HSMMD), 26 W. Martin Luther King Drive, Cincinnati, Ohio-45268.

Project Communications Champion: Diana Cutt, ORD/CESER/Technical Support Coordination Division (TSCD), U.S. EPA Region 2, 290 Broadway, New York-10007.

Regional Lead (s): Ira-Perry Katz, Superfund and Emergency Management Division (SEMD), U.S. EPA Region 2, 290 Broadway, New York-10007.

ORD Team: David Gwisdalla, ORD/CESER/TSCD 26 W. Martin Luther King Drive, Cincinnati, Ohio-45268; Franklin Alvarez, HSMMD, 26 W. Martin Luther King Drive, Cincinnati, Ohio-45268.

Regional Science Liaison: Mindy Pensak, U.S. EPA Region 2, 290 Broadway, New York-10007.

Disclaimer

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Questions concerning this document or its application should be addressed to:

Vasudevan Namboodiri, Ph.D.
Homeland Security and Materials Management Division (HSMMD)
Center for Environmental Solutions and Emergency Response (CESER)
Office of Research and Development (ORD)

U.S. Environmental Protection Agency
26 W. Martin Luther King Dr. Cincinnati, OH 45268,
Email: Namboodiri.Vasudevan@epa.gov

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List of Abbreviations

AOP	advanced oxidation process
APTIM	APTIM Federal Services LLC
ATSDR	Agency for Toxic Substances and Disease Registry
AWBERC	Andrew W. Breidenbach Environmental Research Center
CESER	Center for Environmental Solutions and Emergency Response
ETSC	Engineering Technical Support Center
EPA	Environmental Protection Agency
EQ	equilibration tank
FID	flame ionization detector
GAC	granular activated carbon
GC/MS	gas chromatograph(y)/mass spectrometer (try)
gal	gallon(s)
gpm	gallons per minute
h	hour
H_2O_2	hydrogen peroxide
HASP	Health and Safety Plan
HSMMD	Homeland Security and Materials Management Division
IS	internal standard
ITRC	Interstate Technology & Regulatory Council
L	liter(s)
Lpm	liter(s) per minute
MCL	maximum contaminant level
µg	microgram(s)
mg	milligram(s)
MSD	mass selective detector
NJDEP	New Jersey Department of Environmental Protection
NJ	New Jersey
NPL	National Priorities List
NYSDEC	New York State Department of Environmental Conservation
ORD	Office of Research and Development
O_3	ozone
Pegasus	Pegasus Technical Services, Inc.
PEL	permissible exposure limits
PFD	process flow diagram
POET	point of entry treatment

ppb	part(s) per billion
ppt	part(s) per trillion
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RARE	Regional Applied Research Effort
SEMD	Superfund and Emergency Management Division
SOP	standard operating project plan
SPME	Solid Phase Microextraction
T&E	Test and Evaluation Facility
TSCD	Technical Support Coordination Division
TCA	1,1,1-trichloroethane
THF	tetrahydrofuran
UV	ultraviolet

Acknowledgments

The objective of this project was to demonstrate the ORD's in-house developed cost-effective small-scale Point of Entry (POET) advanced oxidation treatment technology at the Williams Property Superfund site in New Jersey (NJ) with the help of the New Jersey Department of Environmental Protection (NJDEP). This data summary was prepared for the EPA, ORD, CESER, Cincinnati, Ohio, by APTIM Federal Services LLC (APTIM) under Contract No. 68HERC19D0009, Task Order No. 68HERC21F0049. This field demonstration project was supported by EPA Region 2 and ORD's Engineering Technical Support Center (ETSC) through the Technical Support Coordination Division (TSCD). Contributions of the following individuals and organizations to the development of the information provided in this document are acknowledged:

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APTIM Federal Services LLC (Contractor)

Mr. E. Radha Krishnan, P.E., APTIM Program Manager
Mr. Donald A. Schupp, P.E., APTIM Project Leader
Brindha Murugesan
Eric Weaver
Lee Heckman
Dave Elstun
Gary Lubbers
John Brannon
John Brossart.

Pegasus Technical Services (Sub-Contractor)

Dr. Raghuraman Venkatapathy, 1,4-dioxane method development and analyses (using a gas chromatograph/mass spectrometer (GC/MS)) on water samples.

NJDEP

Erin Husta, field test project manager for NJDEP.

Executive Summary

The Williams Property is a 5.6-acre Superfund site in Cape May County, NJ. In August 1979, approximately 150 drums of liquid chemical wastes and sludge were emptied on the site, contaminating soil and groundwater with hazardous chemicals, including 1,4-dioxane. The site currently has a groundwater extraction and treatment system using granular activated carbon (GAC) to treat up to 75 gallons per minute (gpm) of groundwater.

1,4-Dioxane is a likely human carcinogen and has been found in groundwater and drinking water supplies throughout the United States. Historically, 90% of 1,4-dioxane production was used as a stabilizer in chlorinated solvents such as 1,1,1-trichloroethane (TCA). The physical and chemical properties and behavior of 1,4-dioxane create challenges for its characterization and treatment. The compound is highly mobile and does not readily biodegrade in the environment.

1,4-Dioxane is a high-priority chemical in EPA Region 2 due to its widespread occurrence throughout the region and the low regulatory limits established by the NJDEP. As of 2014, there were 544 detections of TCA (a common co-contaminant of 1,4-dioxane) in groundwater throughout NJ; however, these numbers likely underestimate the occurrence of 1,4-dioxane in groundwater across the state. The New York State Department of Environmental Conservation (NYSDEC) is also working with EPA Region 2 to sample for 1,4-dioxane in groundwater at 725 remedial program sites across the state of New York. The preliminary data indicate that levels exceeding the proposed EPA National Primary Drinking Water Regulation[1] Maximum Contaminant Level (MCL) of 1 µg/L occurred at 174, or 24%, of the sites. NYSDEC is currently evaluating a MCL recommendation of 1 µg/L for 1,4-dioxane from the New York Drinking Water Quality Council. If adopted, this MCL would be the nation's most stringent drinking water standard for 1,4-dioxane.

The objective of this project was to conduct a field evaluation of an advanced oxidation assisted 1,4-dioxane treatment technology for treating 1,4-dioxane-contaminated groundwater at the Williams Property Superfund site in NJ. The tests were begun in October 2022 and continued until March 2023.

The treatment system was originally designed, installed, and tested at the EPA Test & Evaluation (T&E) Facility in Cincinnati, OH. It was then shipped to NJ and installed at the Williams Property in September 2022. A 10-gpm slipstream of the groundwater was diverted from the main treatment system to the test system. In its final installed configuration, untreated

[1] National Primary Drinking Water Regulations | US EPA

groundwater from the Williams Property had an average influent 1,4-dioxane concentration of 13.9 µg/L. Treated effluent from the 1,4-dioxane treatment system had an average 1,4-dioxane concentration of 0.86 µg/L. This field study is only to demonstrate that simple treatment system using hydrogen peroxide and ozone called peroxonation will be an easy solution to mitigate the 1,4-dioxane contamination issues in water. This study is specifically designed for treating trace quantities of 1,4 dioxane that may not get removed by regular treatment approaches and GAC filter. It is conducted only for a short period of time, which may not capture all the treatment aspects such as overall treatment cost, influence of other contaminants, and long-term system performance and maintenance requirements.

1.0 Introduction

1.1 Background/Regional Problem

The Williams Property is a 5.6-acre Superfund site in Middle Township, Cape May County, NJ. It is located less than three miles southeast of the Timber Beaver Swamp Fish and Wildlife Management Area, a major aquifer recharge zone, and is bordered by prime wetlands habitats. The nearest surface water is approximately 400 feet northeast of the site in the form of water-filled sand and gravel pits. The nearest natural stream is Deep Creek, which is approximately 3,000 feet southeast of the site (USEPA, 2023; https://cumulis.epa.gov/supercpad/SiteProfiles/index.cfm?fuseaction=second.Cleanup&id=0200678#bkground, last accessed 3/16/2023).

In August 1979, approximately 150 drums of liquid chemical wastes and sludge were emptied on the site, contaminating soil and groundwater with hazardous chemicals, including 1,4-dioxane. Initial actions to protect human health and the environment included removal of approximately 1200 cubic yards of contaminated soil and sludge as well as provision of public water to approximately 140 homes and businesses that were potentially affected by the site. It currently has a groundwater extraction and treatment system using granular activated carbon (GAC) to treat up to 75 gallons per minute (gpm) of groundwater.

1,4-Dioxane is a likely human carcinogen and has been found in groundwater and drinking water supplies throughout the United States (USDHHS, 2021). Historically, 90% of 1,4-dioxane was produced for use as a stabilizer in chlorinated solvents such as 1,1,1-trichloroethane (TCA); however, 1,4-dioxane was also an unintended contaminant of chemical ingredients used in consumer products including bubble bath, shampoo, laundry detergent, soap, skin cleanser, adhesives, and antifreeze (MDH, 2013 and 2025). The physical and chemical properties and behavior of 1,4-dioxane create challenges for its characterization and treatment. The compound is highly mobile and does not readily biodegrade in the environment. Synonyms for 1,4-dioxane are dioxane, *p*-dioxane, diethylene ether, diethylene dioxide, and glycol ethylene ether (EPA, 2006). 1,4-Dioxane has been identified as an emerging contaminant of concern, having been detected in both EPA Superfund sites and public water supplies throughout the United States (EPA, 2017).

1,4-Dioxane has been found in at least 31 of the 1,689 current or former National Priorities List (NPL) sites (ATSDR, 2012), and 1,4-dioxane is a high-priority chemical in EPA Region 2 due to its widespread occurrence throughout the region and the low regulatory limits established by the NJDEP. The NYSDEC is currently evaluating an EPA National Primary Drinking Water

Regulation[2] MCL recommendation of 1 µg/L for 1,4-dioxane from the New York Drinking Water Quality Council. If adopted, this MCL would be the nation's most stringent drinking water standard for 1,4-dioxane.

Commonly used treatment technologies to remove 1,4-dioxane and other organic chemicals from water include advanced oxidation processes (AOPs), granular activated carbon (GAC), and synthetic media. AOPs are a group of technologies that use the highly reactive hydroxyl radical to destructively remove organic contaminants (ITRC, 2021). The most-used AOPs are those that involve ozone (O_3), hydrogen peroxide (H_2O_2), and ultraviolet (UV) light (Broughton et al., 2019; Elkacmi et al., 2019).

The blending of O_3 and H_2O_2 is called peroxonation and is a powerful oxidation process for treating 1,4-dioxane in water. Hydroxyl radicals are formed when O_3 and H_2O_2 are added to water simultaneously. Peroxonation is also an established technology and has shown effective reduction of 1,4-dioxane concentrations to less than 2 µg/L in California (Mohr et al., 2010). The main disadvantages with these systems are bromate formation in bromide-containing waters and the need to destroy the unreacted O_3 and H_2O_2.

1.2 Research Objectives

The objective of this EPA Regional Applied Research Effort (RARE) collaborative research project was to conduct a field evaluation of a cost-effective, low-maintenance technology for treating 1,4-dioxane-contaminated groundwater at the Williams Property Superfund site in NJ. The tests were initiated in October 2022 and continued through March 2023. The project: 1) tested the efficacy of a promising low-cost advanced oxidation technology — an O_3/UV system — in treating 1,4-dioxane levels to the NJDEP criterion of 0.4 µg/L; 2) evaluated the monitoring and maintenance requirements for an O_3/UV treatment system design; and 3) provided recommendations for the best treatment configuration for small-scale treatment systems.

While1,4-Dioxane is a high-priority chemical in EPA Region 2, this research is relevant to other EPA regions since 1,4-dioxane has been found in groundwater and drinking water supplies throughout the U.S. Several states have established groundwater criteria as shown in Table 1.

[2] National Primary Drinking Water Regulations | US EPA

Table 1. State groundwater criteria

State	1,4-Dioxane Guidance (µg/L)	State	1,4-Dioxane Guidance (µg/L)
Alaska	4.6	Mississippi	6.09
California	1.0	New Hampshire	0.25
Colorado	0.35	New Jersey	0.4
Connecticut	3.0	North Carolina	3.0
Delaware	6.0	Pennsylvania	6.4
Florida	3.2	Texas	9.1
Indiana	7.8	Vermont	3.0
Maine	4.0	Washington	0.438
Massachusetts	0.3	West Virginia	6.1

This project aimed to determine the treatment effectiveness of advanced UV oxidation to achieve 1,4-dioxane reduction to meet the regulatory level needs. The study evaluated 1,4-dioxane contaminated source water and effectiveness of the treatment system. Water quality monitoring technologies were used before and after treatment to ensure the system was effective. EPA health and safety plans (HASPs) and quality assurance project plans (QAPPs) were designed to ensure that the experimental results and project findings collected data with established quality for their intended use.

The treatment system was originally designed, installed, and tested at the EPA Test & Evaluation (T&E) Facility by APTIM Federal Services LLC (APTIM) under EPA Contract No. 68HERC19D0009, Task Order No. 68HERC21F0049 in Cincinnati, OH (Namboodiri et al. 2023). The system was then shipped to NJ and installed at the Williams Property in September 2022. A 10-gpm slipstream of the groundwater was diverted from the main treatment system to the test system.

Operation of the treatment system was performed by Handex Consulting & Remediation, LLC, while operating the full-scale groundwater extraction/treatment system at the site. Pegasus Technical Services, Inc. (Pegasus) was subcontracted to APTIM to perform 1,4-dioxane analysis and provide technical support.

1.3 Report Organization

This report is organized in the following sections. This section (Section 1.0) addresses the background and purpose of the study, introduces the project team, and provides an outline of the report. Section 2.0 describes the materials and methods including the treatment system involved in treating 1,4-dioxane-contaminated groundwater and the analytical methods used in the study. Section 3.0 presents the results of treatment system evaluation; Section 4.0 gives a summary and conclusion of the study and Section 5.0 provides a list of references.

2.0 Materials and Methods

2.1 Test Water

Test water for this study was 1,4-dioxane-contaminated groundwater from the Williams Property in NJ. The Williams Property currently has a groundwater extraction and treatment system using granular activated carbon (GAC) to treat up to 75 gpm of groundwater as shown in Figure 1. Groundwater was extracted from the site using the existing groundwater extraction system, and a 10-gpm slipstream of the groundwater was piped to the treatment system for evaluation.

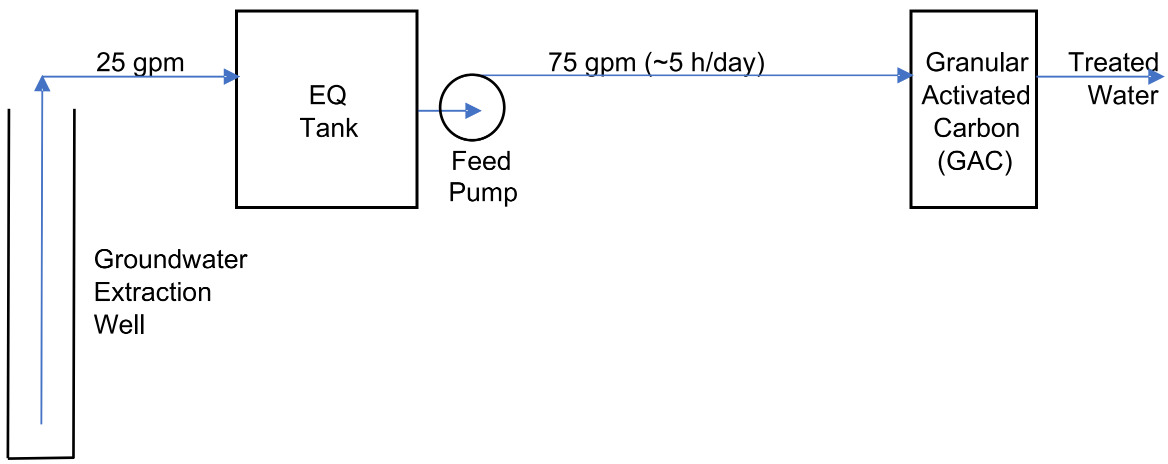

Figure 1. Current Williams Property groundwater treatment system

2.2 1,4-Dioxane Treatment System

The groundwater treatment system used in this study included O_3 and H_2O_2 (Namboodiri et al. 2023). Table 2 lists the equipment used to assemble the treatment system. The process flow diagram (PFD) for the treatment system is shown in Figure 2.

Water enters the system and passes through a flow switch. The flow switch, through a control box, activates the oxygen generator, which then activates the O_3 generator and produces O_3. After passing through the flow switch, the water goes through a flowmeter, which controls the feed rate of a pump that injects H_2O_2 into the water at a concentration of approximately 3.5 mg/L. Following the H_2O_2 injection, the manufactured O_3 is pulled into the water through a venturi injector. An O_3 concentration of approximately 4.5 mg/L is measured in the water. The water then passes through a flash reactor and into a 25-gallon contact tank. Undissolved O_3 from the water leaves the contact tank and passes through an O_3 destruct unit. An O_3 monitor measures the O_3 in the ambient air to ensure the O_3 concentration does not become hazardous. The Occupational Safety and Health Administration Permissible Exposure Limit (PEL)[3] for O_3 is 0.1 ppm. After the contact tank, the water passes through a GAC filter to remove any remaining H_2O_2, O_3, and organic chemicals. Initial testing used an air dryer instead of an oxygen generator and a 15-gallon contact tank in place of a 25-gallon contact tank, but these were replaced to improve the system's performance.

During our technology development phase, we found that this peroxonation treatment system was very effective in destroying 1,4-dioxane in water. By using a combination of approximately 3.5 mg/L H_2O_2 and 5 mg/L O_3, water flow rates up to 10 gpm containing up to 200 µg/L 1,4-dioxane were treated. Influent 1,4-dioxane concentrations of approximately 10 µg/L, 20 µg/L, 80 µg/L, and 180 µg/L were reduced to effluent 1,4-dioxane concentrations of approximately 0.4 µg/L, 0.7 µg/L, 5 µg/L, and 10 µg/L, respectively, when treated at flow rates of approximately 10 gpm (Namboodiri et al. 2023).

Figure 3 shows the H_2O_2 injection system. Figure 4 shows the O_3 system.

Table 2. Treatment system equipment

Component	**Model and Source**	**Price**
H_2O_2 Injection	Clean Water Store (www.cleanwaterstore.com) H_2O_2 Proportional Flow Well Water J-PRO-22 - Tank size: 5 gallons - Water meter size: ¾” - Injection tee: ¾”	$913
Flow Switch	Oxidation Technologies (www.oxidationtech.com) Control Box with Flow Switch	$325
Oxygen Generator	Oxidation Technologies (www.oxidationtech.com) MAX-5 Oxygen Generator	$1360
O_3 Generator	Oxidation Technologies (www.oxidationtech.com) WT-10 O_3 Water System - Venturi injector Model 684 - Contact tank 25 gallons - Static mixer Model 73-NK	$4480

[3] https://www.osha.gov/annotated-pels

Component	Model and Source	Price
O_3 Destruct Device	Oxidation Technologies (www.oxidationtech.com) CDU-30 O_3 Destruct Device - Wall bracket - Water trap - Heater element	$895
O_3 Monitor	Oxidation Technologies (www.oxidationtech.com) C-30ZX O_3 Monitor	$536

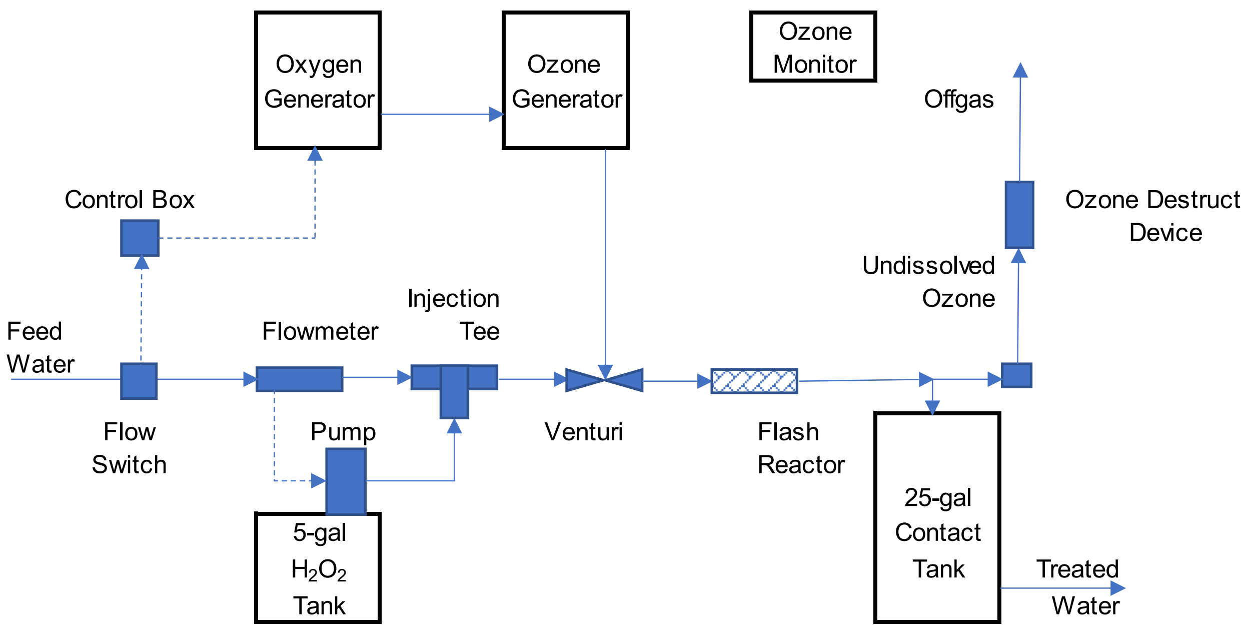

Figure 2. 1,4-Dioxane treatment process flow diagram

Figure 3. H_2O_2 injection system

Figure 4. O_3 system (air dryer, oxygen generator, O_3 generator, contact tank)

2.3 Analytical Methods

Grab samples were collected from two points in the system: influent and effluent. 1,4-Dioxane samples were shipped to the EPA T&E Facility and transported to the Andrew W. Breidenbach Environmental Research Center (AWBERC) where they were extracted and analyzed by Pegasus. All collected samples were analyzed for 1,4-dioxane. Samples were collected headspace-free in 40-mL amber vials and stored at 4±2 ºC until analyzed.

Samples were analyzed for 1,4-dioxane by adding approximately 10-mL of the aqueous sample to a 20-mL autosampler vial and spiking with a surrogate mix and internal standard (IS). Initial analysis was carried out using d_8-tetrahydrofuran as the IS, and d_8-1,4-dioxane, d_3-methyl tert-butyl ether, 4-bromofluorobenzene and d_4-1,2-dichlorobenzene as the surrogate standards (Spex Certiprep, Metuchen, NJ). Based on matrix effects encountered, d_8-1,4-dioxane (Spex Certiprep, Metuchen NJ) was instead used as the IS, and d_8-tetrahydrofuran added as a fourth surrogate, and the final concentration was determined using isotopic dilution. The samples were then quantified using an Agilent 7890A Gas Chromatograph (GC) with a 5975C Tripple Axis Mass Selective Detector (MSD) with Triple Axis Detector (Agilent Technologies, Santa Clara, CA) and CombiPal autosampler (CTC Analytics, Zwingen Switzerland) following EPA Method 524.3 modified to perform headspace analysis instead of purge and trap. With the modified method, the autosampler heats the sample-containing vials to 90˚C for 30 minutes prior to analysis. An aliquot of air (adjustable from 250 µL to 2500 µL) drawn from the headspace was injected into the GC/MS. The data generated in this study will be checked for the acceptance criteria as described in the Quality control requirements mentioned in the Appendix A of this report. In our previous report on 1,4-dioxane (Namboodiri, 2023), we compared EPA method 522 vs EPA Method 524.3 (modified) and recommend latter as a robust method that can be used to determine 1,4-dioxane concentrations even at low levels (low ppt), while minimizing sample processing from days to few minutes. This method uses concentrating samples using solid phase extraction (SPE) that potentially lead to lower detection limits.

3.0 Results

3.1 Initial Installation (December 2022 – February 2023)

The 1,4-dioxane treatment system was connected to the Williams Property groundwater extraction/treatment system by using a 10-gpm slipstream of the untreated groundwater as shown in Figure 5. The effluent from the 1,4-dioxane treatment system was then pumped back to the untreated groundwater before it entered the GAC filter as shown in Figure 5.

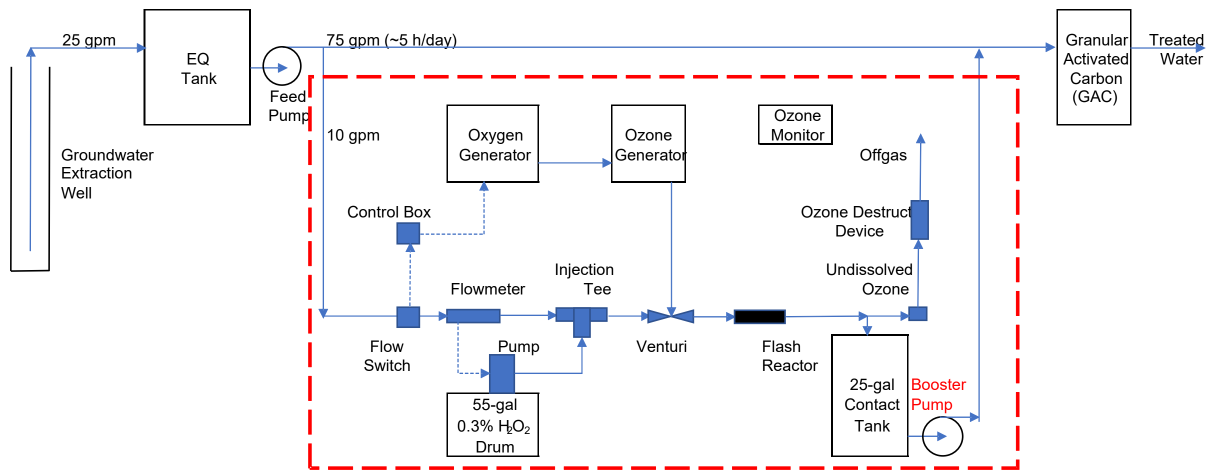

Figure 5. Initial installation of the 1,4-dioxane treatment system at the Williams Property

After shakedown testing in October – November 2022, testing of the 1,4-dioxane treatment system began in December 2022 and continued through February 2023. The system was operated continuously 8 am to 5 pm. Table 3 shows the influent and effluent samples from the 1,4-dioxane treatment system. The average 1,4-dioxane influent concentration was 12.69 µg/L with a standard deviation of 1.73, and the average 1,4-dioxane effluent concentration was 1.97 µg/L with a standard deviation 0.80. This was due to ozone injection variations occurred due to back-pressure in the system.

Table 3. 1,4-Dioxane treatment system concentrations for the initial 1,4-dioxane treatment system installation

Date	Sample collection Time	Influent (µg/L)	Effluent (µg/L)
12/13/2022	13:30	11.59	2.78
12/13/2022	13:45	11.90	3.11
12/13/2022	14:00	11.69	2.70
12/13/2022	14:45	9.04	1.49
12/13/2022	15:00	9.73	1.79

Date	Sample collection Time	Influent (µg/L)	Effluent (µg/L)
12/13/2022	15:15	11.19	1.39
12/16/2022	9:00	11.02	0.52
12/16/2022	9:30	11.49	0.52
12/16/2022	9:45	10.71	1.07
12/16/2022	11:00	12.87	0.74
12/16/2022	11:30	12.16	0.68
12/16/2022	11:45	10.75	1.11
12/20/2022	9:00	10.95	1.10
12/20/2022	9:30	10.68	0.86
12/20/2022	9:45	10.31	0.80
12/20/2022	11:00	11.10	0.79
12/20/2022	11:30	11.02	0.83
12/20/2022	11:45	11.11	1.00
1/10/2023	9:30	13.54	3.81
1/10/2023	10:00	13.91	2.49
1/10/2023	10:30	13.06	1.67
1/10/2023	11:00	11.13	1.46
1/10/2023	11:30	13.60	1.20
1/10/2023	12:00	15.27	1.59
1/10/2023	12:30	14.18	1.21
1/12/2023	9:00	13.85	1.68
1/12/2023	9:30	16.77	1.61
1/12/2023	10:00	14.58	1.33
1/12/2023	10:30	15.63	1.35
1/12/2023	11:00	15.28	1.36
1/12/2023	11:30	13.05	1.90
1/12/2023	12:00	14.95	1.41
1/12/2023	12:30	12.06	1.54
1/12/2023	13:00	12.11	1.92
1/12/2023	13:30	15.09	1.46
2/14/2023	12:30	12.85	2.85
2/14/2023	12:45	12.54	2.80
2/14/2023	13:00	11.95	2.36
2/14/2023	13:15	12.08	2.75
2/14/2023	13:30	12.18	2.28
2/14/2023	13:45	12.17	2.60
2/14/2023	14:00	11.99	2.74
2/14/2023	14:15	11.73	1.96
2/15/2023	12:30	12.74	3.16
2/15/2023	12:45	12.63	2.99
2/15/2023	13:00	11.53	2.56
2/15/2023	13:15	12.01	1.70
2/17/2023	11:00	13.31	2.92
2/17/2023	11:15	13.67	2.53
2/17/2023	11:30	13.84	2.74
2/17/2023	11:45	12.89	2.49

Date	Sample collection Time	Influent (µg/L)	Effluent (µg/L)
2/17/2023	12:00	12.95	2.74
2/17/2023	12:30	12.57	2.57
2/17/2023	13:00	13.30	2.81
2/17/2023	13:30	13.06	2.52
2/17/2023	14:00	13.34	1.75
2/20/2023	10:00	12.35	2.97
2/20/2023	10:15	11.60	1.53
2/20/2023	10:30	12.04	1.79
2/20/2023	10:45	17.83	2.58
2/20/2023	11:00	11.71	2.13
2/20/2023	11:30	11.54	2.28
2/20/2023	12:00	13.50	2.72
2/20/2023	12:30	10.92	2.13
2/20/2023	13:00	17.37	3.15
2/20/2023	13:30	16.01	3.36
2/20/2023	14:00	12.89	1.53
Average		**12.69**	**1.97**
Std. Dev.		**1.735**	**0.803**

3.2 Revised Installation (March 2023)

In March 2023, the installation configuration was changed to move the booster pump location from the end of the 1,4-dioxane treatment system to a position between the H_2O_2 and O_3 injection points. Also, the discharge of the system was directed directly to the sewer rather than rejoining the untreated groundwater prior to the GAC filter. These changes improved the H_2O_2 mixing with the groundwater, eliminated O_3 entering and corroding the booster pump, and improved the O_3 injection in the system due to back pressure. This configuration is shown in Figure 6.

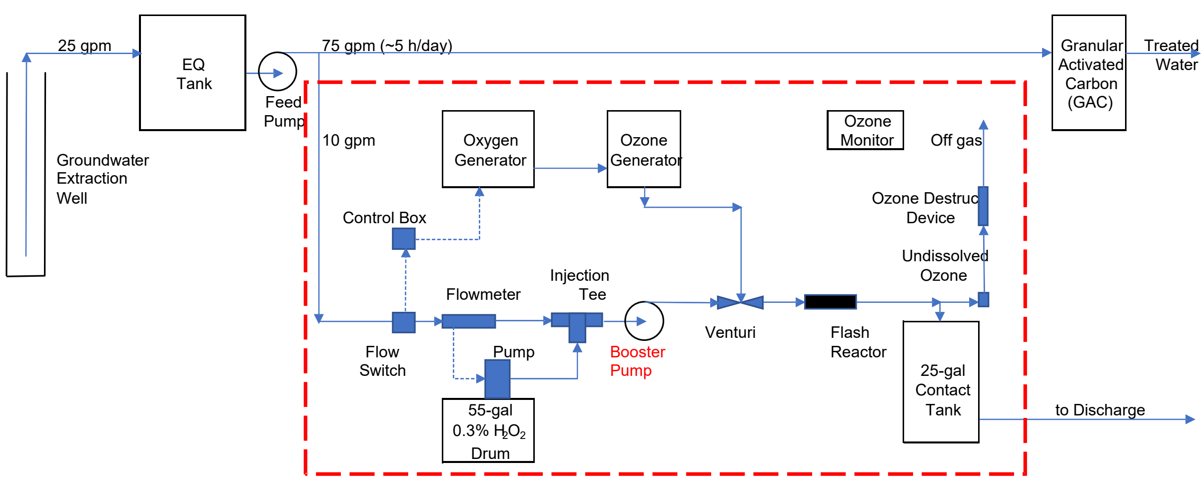

Figure 6. Revised installation of the 1,4-dioxane treatment system at the Williams Property with the booster pump moved and a separate discharge

Table 4 shows the influent and effluent samples from the 1,4-dioxane treatment system after the installation configuration was revised. The average 1,4-dioxane influent concentration was 13.85 µg/L with a standard deviation of 1.93, and the average 1,4-dioxane effluent concentration was 0.86 µg/L (~94% reduction) with a standard deviation of 0.37 in the revised configuration.

Table 4. 1,4-Dioxane treatment system concentrations for the revised 1,4-Dioxane treatment system installation

Date	Sample Collection Time	Influent (µg/L)	Effluent (µg/L)
3/9/2023	12:00	13.36	1.17
3/9/2023	12:20	12.86	1.29
3/9/2023	12:40	11.10	1.14
3/9/2023	13:00	13.31	1.21
3/9/2023	13:20	10.45	0.87
3/9/2023	13:40	12.90	0.74
3/9/2023	14:00	12.43	0.74
3/14/2023	8:00	11.43	0.59
3/14/2023	8:30	10.79	0.49
3/14/2023	9:00	19.33	0.46
3/14/2023	9:30	12.61	0.46
3/14/2023	10:00	11.74	0.47
3/14/2023	11:00	13.47	0.46
3/14/2023	12:00	12.74	0.45
3/14/2023	13:00	12.74	0.38
3/15/2023	9:00	14.50	0.73
3/15/2023	9:30	14.97	0.66
3/15/2023	10:00	12.79	0.48
3/15/2023	10:30	15.23	2.11
3/15/2023	11:00	15.75	1.06
3/15/2023	12:00	16.34	0.81
3/15/2023	13:00	14.83	0.90
3/15/2023	14:00	15.66	0.64
3/15/2023	14:30	17.30	0.58
3/16/2023	8:30	14.05	0.79
3/16/2023	9:00	13.07	1.22
3/16/2023	9:30	13.76	1.14
3/16/2023	10:00	15.70	1.18
3/16/2023	10:30	13.44	0.90
3/16/2023	11:00	15.11	1.44
3/16/2023	12:00	14.60	0.91
3/16/2023	13:00	14.89	0.91
Average		**13.85**	**0.86**

Date	Sample Collection Time	Influent (µg/L)	Effluent (µg/L)
Std. Dev.		**1.93**	**0.37**

At present, we are in the process of developing automated and largescale (100-1200gpm) mobile versions of the technology through industrial collaboration for long term use for various water treatment and reuse applications. A 500gpm system is being operated in a utility for more than year solving their water treatment issues. Details of scaling up and field applications will be summarized in a future report.

4.0 Conclusions

The objective of this project was to conduct a field evaluation of a cost-effective advanced oxidation treatment technology for treating 1,4-dioxane-contaminated groundwater at the Williams Property Superfund site in NJ. The tests were initiated in October 2022 and continued through March 2023.

After shakedown testing in October – November 2022, testing of the 1,4-dioxane treatment system began in December 2022 and continued through February 2023. During this period, the average 1,4-dioxane influent concentration was 12.7 µg/L, the average 1,4-dioxane effluent concentration was 2.0 µg/L, and the 1,4-dioxane effluent concentration was consistently below 3 µg/L (average 84% reduction with a standard deviation of 0.803).

In March 2023, the installation configuration was changed to move the booster pump location from the end of the 1,4-dioxane treatment system to between the H_2O_2 and O_3 injection points. Also, the discharge of the system was directed directly to the sewer rather than rejoining the untreated groundwater prior to the GAC filter. These changes improved the H_2O_2 mixing in the groundwater, eliminated O_3 entering and corroding the booster pump, and improved the O_3 injection in the system. The revised configuration of the system could reduce influent 1,4-dioxane concentration averaged 13.9 µg/L to an average effluent concentration of 0.86 µg/L (average 94% reduction with a standard deviation of 0.37).

The study shows that hydrogen peroxide and ozone combination is an effective solution to mitigate the 1,4-dioxane contamination issues in water. This study is specifically designed for treating trace quantities of 1,4 dioxane that may not get removed by regular treatment approaches and GAC filter. The demonstration is conducted only for a short period of time, which may not capture all the treatment aspects such as overall treatment cost, influence of other contaminants, energy consumption, long-term system performance and maintenance requirements. This treatment is designed only for final polishing not for overall treatment of the water. In this real-world scenario, we were able to demonstrate the potential of the technology even for untreated ground water with a 1,4-dioxane reduction of ~94%. More detailed field study is recommended for the evaluation of overall performance of the system compared to the existing treatment in removing other contaminants.

Future directions of this continuous flow water treatment technology will include treatment of other trace organic chemicals such as pesticides and pharmaceuticals. We are also engaged in integrating this technology with other treatment technologies such as ceramic filtration, electro-oxidation and ultraviolet treatment technology for developing mobile units for various water treatment applications.

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Appendix A – Summary of the Quality Assurance/Quality Control (QA/QC) Measures

A.1 Introduction

An important aspect of technology testing is the QA/QC procedures and requirements developed. Careful adherence to the procedures detailed in the Quality Assurance Project Plan (QAPP) enables researchers to evaluate the performance of the treatment system being evaluated for 1,4-dioxane destruction. The primary measures of evaluation for data quality were accuracy, precision, completeness, and representativeness.

Water samples for the field evaluation were collected from the Williams Property in NJ. Analysis of the samples for 1,4-dioxane was performed at the EPA AWBERC. System performance evaluation testing and laboratory activities were conducted by APTIM, Handex, and Pegasus in accordance with the provisions of the EPA *Quality Requirements for Measurement Projects* (U.S. EPA, 2008).

A.2 Analytical Procedures

APTIM, Handex, and Pegasus staff conducted the performance evaluation tests following an EPA-approved QAPP (APTIM, 2020) that was created specifically for these evaluations. Pegasus staff conducted the 1,4-dioxane analyses. Analytical methods for 1,4-dioxane analyses are presented in Table A-1.

Table A-1. Measurements and Analytical Methods

Measurement	Analytical Method Standard Operating Procedure (SOP)
1,4-Dioxane (Headspace)	EPA Method 524.3 (modified)

A.3 Sample Handling

Samples collected by Handex were labeled with unique identification numbers in the format specified in the EPA-approved QAPPs. Samples were shipped to the EPA T&E Facility in hard-sided coolers with ice and transferred by APTIM from the T&E Facility to AWBERC for 1,4-dioxane analyses by Pegasus. All samples were analyzed within sample holding times identified in the QAPPs.

A.4 Sample QA/QC

The calibration of analytical instruments and the analyses of parameters complied with the QA/QC provisions of the EPA-approved QAPP used in this evaluation. Sample volumes, preservation, and holding times are shown in Table A-2. Laboratory QA/QC checks for 1,4-dioxane analyses are shown in Table A-3.

The APTIM QA/QC requirements specified in the referenced methods (Table A-1) are compliant with those stated in the EPA-approved QAPPs and based on EPA published methods for 1,4-dioxane.

Table A-2. Sample Volumes, Preservation, and Holding Times

Measurement	Sample Container	Volume of Sample	Preservation	Holding Time
1,4-Dioxane (Headspace)	VOA vial	40 mL	pH<2; <6 ºC	14 days

VOA: volatile organic analysis

Table A-3. QA/QC Checks

Measurement	QA/QC Check	Frequency	Acceptance Criteria	Corrective Action
1,4-Dioxane [EPA Method 524.3 (modified)]	Calibration	At the beginning of sequence or after CCC failure	$R^2 \geq 0.99$	Investigate problem. Prepare new calibration standards.
	Laboratory control sample (LCS)	Following calibration	±25% of the true value	Recalibrate.
	Continuing calibration check (CCC)	Beginning/end of each batch and after every 10 samples	±25% of the true value	Evaluate data for usability. Recalibrate, reanalyze affected samples.
	Sample matrix blank	One per batch	< the lowest sample concentration for each analyte	Reanalyze. Evaluate data for usability.
	Sample duplicates	One per batch	±25% Relative percent difference	Reanalyze. Evaluate data for usability.
	Laboratory fortified sample matrix	One per batch	±50% of the true value	Reanalyze. Evaluate data for usability.
	Surrogate	Add to each sample	±30% of the true value	Reanalyze. Evaluate data for usability.
	Internal standard	Add known quantity to each sample prior to reconstituting	Area must be within ±50% of the average peak area in the initial calibration	Reanalyze. Evaluate data for usability.

Measurement	QA/QC Check	Frequency	Acceptance Criteria	Corrective Action
	Standard reference material (standard reference material, if available)	One per sequence	±35% of the certified value for 70% of the analytes	Reanalyze. Evaluate data for usability.

A.5 Test System QA/QC

Samples were collected according to the schedule provided in the EPA-approved QAPP. Field duplicate samples were collected to verify the homogeneity of test water concentrations. No significant variations were observed for the field duplicate samples based on accuracy and precision. Duplicate sample analyses are included in Table A-4. Standard recoveries are included in Table A-5. Surrogate recoveries are included in Table A-6, and laboratory fortified blank (LFB)/laboratory fortified matrix (LFM) spike recoveries are included in Table A-7.

Table A-4. Duplicate Sample Analysis for 1,4-Dioxane

Date	Method	Units	Sample 1	Sample 2	RPD[1] (%)
10/23/2022	In-house Headspace	ppt	2047.22	2030.09	0.84
10/23/2022	In-house Headspace	ppt	2034.60	1979.72	2.73
10/23/2022	In-house Headspace	ppt	1870.03	1847.52	1.21
10/23/2022	In-house Headspace	ppt	1905.16	1893.83	0.60
10/23/2022	In-house Headspace	ppt	1861.02	1799.69	3.35
10/23/2022	In-house Headspace	ppt	2359.57	2329.27	1.29
11/02/2022	In-house Headspace	ppt	13404.70	13149.71	1.92
11/02/2022	In-house Headspace	ppt	18391.82	18266.69	0.68
12/25/2022	In-house Headspace	ppt	3436.75	3401.52	1.03
12/25/2022	In-house Headspace	ppt	8577.10	8427.56	1.76
03/01/2023	In-house Headspace	ppt	9269.70	9573.53	3.22
03/08/2023	In-house Headspace	ppt	12238.78	12435.69	1.59
03/21/2023	In-house Headspace	ppt	10535.18	11068.30	4.93
03/21/2023	In-house Headspace	ppt	12430.58	12874.91	3.51
03/21/2023	In-house Headspace	ppt	446.02	432.19	3.14
03/21/2023	In-house Headspace	ppt	17301.25	17920.30	3.515
03/21/2023	In-house Headspace	ppt	906.39	882.63	2.65

1. Relative precent difference (RPD) calculated as described in Section A.8.2.

Table A-5. Standard Recoveries for 1,4-Dioxane

Date	Method	Units	Standard	Measured	Recovery (%)
10/13/2022	In-house Headspace	ppt	5000	5068.53	101.37
10/13/2022	In-house Headspace	ppt	5000	5488.06	109.76
10/23/2022	In-house Headspace	ppt	5000	5157.27	103.15

Date	Method	Units	Standard	Measured	Recovery (%)
10/23/2022	In-house Headspace	ppt	5000	5023.43	100.47
10/23/2022	In-house Headspace	ppt	5000	4818.83	96.38
10/23/2022	In-house Headspace	ppt	5000	4815.96	96.32
10/23/2022	In-house Headspace	ppt	5000	4709.22	94.18
11/02/2022	In-house Headspace	ppt	500	525.85	105.17
11/02/2022	In-house Headspace	ppt	500	505.22	101.04
12/25/2022	In-house Headspace	ppt	500	478.40	95.68
12/25/2022	In-house Headspace	ppt	500	463.07	92.61
12/25/2022	In-house Headspace	ppt	500	485.64	97.13
12/25/2022	In-house Headspace	ppt	500	504.90	100.98
12/25/2022	In-house Headspace	ppt	500	495.89	99.18
12/25/2022	In-house Headspace	ppt	500	489.71	97.94
02/24/2023	In-house Headspace	ppt	5000	5374.31	107.50
02/24/2023	In-house Headspace	ppt	5000	4901.17	98.00
02/24/2023	In-house Headspace	ppt	5000	4767.23	95.30
03/01/2023	In-house Headspace	ppt	500	495.22	99.00
03/01/2023	In-house Headspace	ppt	500	519.33	103.90
03/01/2023	In-house Headspace	ppt	500	506.75	101.30
03/01/2023	In-house Headspace	ppt	500	528.12	105.60
03/01/2023	In-house Headspace	ppt	2500	2762.98	110.50
03/01/2023	In-house Headspace	ppt	500	568.38	113.70
03/01/2023	In-house Headspace	ppt	500	503.21	100.60
03/08/2023	In-house Headspace	ppt	5000	5060.40	101.21
03/08/2023	In-house Headspace	ppt	5000	5007.54	100.15
03/08/2023	In-house Headspace	ppt	5000	5053.95	101.08
03/08/2023	In-house Headspace	ppt	5000	4879.04	97.58
03/08/2023	In-house Headspace	ppt	5000	4792.68	95.85
03/08/2023	In-house Headspace	ppt	5000	4401.70	88.03
03/08/2023	In-house Headspace	ppt	5000	4488.91	89.78
03/08/2023	In-house Headspace	ppt	5000	4604.22	92.08
03/08/2023	In-house Headspace	ppt	5000	5175.07	103.50
03/08/2023	In-house Headspace	ppt	5000	4899.91	98.00
03/08/2023	In-house Headspace	ppt	5000	4123.00	82.46
03/21/2023	In-house Headspace	ppt	500	525.68	105.14
03/21/2023	In-house Headspace	ppt	500	557.78	111.56
03/21/2023	In-house Headspace	ppt	500	548.92	109.78
03/21/2023	In-house Headspace	ppt	500	563.36	112.67
03/21/2023	In-house Headspace	ppt	500	502.66	100.53
03/21/2023	In-house Headspace	ppt	500	490.16	98.03

Recovery calculated as described in Section A.8.1.

Table A-6. Surrogate Recoveries for 1,4-Dioxane

Date	Method	Units	Spike	Measured	Recovery (%)
10/13/2022	In-house Headspace	ppt	5000	4602.86	92.06
10/13/2022	In-house Headspace	ppt	5000	4871.70	97.43
10/23/2022	In-house Headspace	ppt	5000	5273.67	105.47
10/23/2022	In-house Headspace	ppt	5000	5087.88	101.76
10/23/2022	In-house Headspace	ppt	5000	5061.51	101.23
10/23/2022	In-house Headspace	ppt	5000	4774.73	95.49
10/23/2022	In-house Headspace	ppt	5000	4369.67	87.39
11/02/2022	In-house Headspace	ppt	500	479.48	95.90
11/02/2022	In-house Headspace	ppt	500	415.96	83.19
12/25/2022	In-house Headspace	ppt	500	438.24	87.65
12/25/2022	In-house Headspace	ppt	500	410.33	82.07
12/25/2022	In-house Headspace	ppt	500	429.29	85.86
12/25/2022	In-house Headspace	ppt	500	434.92	86.98
12/25/2022	In-house Headspace	ppt	500	429.92	85.98
12/25/2022	In-house Headspace	ppt	500	442.37	88.47
02/24/2023	In-house Headspace	ppt	5000	5356.00	107.10
02/24/2022	In-house Headspace	ppt	5000	5508.85	110.20
02/24/2023	In-house Headspace	ppt	5000	5390.27	107.80
03/01/2023	In-house Headspace	ppt	500	433.99	86.80
03/01/2023	In-house Headspace	ppt	500	436.08	87.20
03/01/2023	In-house Headspace	ppt	500	422.16	84.40
03/01/2023	In-house Headspace	ppt	500	418.83	83.80
03/01/2023	In-house Headspace	ppt	2500	2231.27	89.30
03/01/2023	In-house Headspace	ppt	500	446.82	89.40
03/01/2023	In-house Headspace	ppt	500	433.75	86.70
03/21/2023	In-house Headspace	ppt	500	516.77	103.35
03/21/2023	In-house Headspace	ppt	500	490.42	98.08
03/21/2023	In-house Headspace	ppt	500	423.30	84.66
03/21/2023	In-house Headspace	ppt	500	391.63	78.33
03/21/2023	In-house Headspace	ppt	500	397.31	79.46
03/21/2023	In-house Headspace	ppt	500	410.80	82.16

Table A-7. LFB/LFM Spike Recoveries for 1,4-Dioxane

Date	Method	Units	Spike	LFB/LFM	Sample	Recovery (%)
10/23/2022	In-house Headspace	ppt	500	1861.02	2359.57	99.71
10/23/2022	In-house Headspace	ppt	500	1799.69	2329.27	105.92
11/02/2022	In-house Headspace	ppt	5000	13404.70	18391.82	99.74
11/02/2022	In-house Headspace	ppt	5000	13149.71	18266.69	102.34
12/25/2022	In-house Headspace	ppt	5000	3436.75	8577.10	102.81
12/25/2022	In-house Headspace	ppt	5000	3401.52	8427.56	100.52

Recovery calculated as described in Section A.8.1.

A.6 Documentation

Laboratory activities were documented using standardized datasheets, logbooks, and laboratory notebooks. Laboratory data reports were entered into Microsoft™ Excel® spreadsheets. These spreadsheets were used to calculate the mean, standard deviation, and ranges, as applicable.

A.7 Data Review

Calculations performed on a computer were checked initially by the analyst for gross error and miscalculation. The calculations and data were entered into computer spreadsheets were checked by a peer reviewer for accuracy by printing out the calculation or data spreadsheet and checking the calculation by hand or comparing each entry of data with the original.

A.8 Data Quality Indicators

The quality of data generated for this system performance evaluation was established through four indicators of data quality: accuracy, precision, completeness, and representativeness.

A.8.1 Accuracy

Accuracy was quantified as the percent recovery of the parameter in a sample of known quantity. Accuracy was measured through use of certified standards during calibration of an instrument.

Percent Recovery was calculated using the following equation:

For controls:

$$\%R = (M/K)*100\%$$

For matrix spike:

$$\%R = [(Xs-Xu)/K]*100\%$$

where

R = percent recovery
M = Measured analyte concentration
K = Known analyte/spike concentration
Xs = Measured concentration of analyte in spiked sample
Xu = Measured concentration of analyte in unspiked sample.

A.8.2 Precision

Precision refers to the degree of mutual agreement among individual measurements and provides an estimate of random error. Precision of duplicate analyses was measured using the following equation to calculate RPD:

$$RPD = \left| \frac{S_1 - S_2}{S_1 + S_2} \right| \times 200$$

where:

S_1 = sample analysis result; and

S_2 = sample duplicate analysis result.

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

$$RSD = (s/y_{ave}) \ x \ 100\%$$

where:

RSD = relative standard deviation (%)

s = standard deviation

y_{ave} = mean of the replicate analyses

Standard deviation is defined as follows:

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

where:

s = standard deviation

y_i = measured value of the i^{th} replicate

y_{ave} = mean of the replicate measurements

n = number of replicates

A.8.3 Completeness

Completeness is a measure of the relative number of analytical data points that meet all the acceptance criteria for accuracy, precision, and additional criteria required by the specific analytical methods used. The goal is that sufficient amounts of valid data will be generated to satisfy the quality assurance conditions. Completeness was expressed as a percentage, as follows:

Percent Completeness = (number of valid data points)/(expected number of data points) x 100%

The completeness goal for this study was 100%.

A.8.4 Representativeness

Representativeness describes the degree to which sample data accurately and precisely represents a characteristic of the material being measured. Representativeness is a qualitative term that is evaluated to determine whether field measurements were made, and physical samples were collected, in such a manner that the resulting data appropriately reflect the media and phenomena measured or studied.

Representativeness was determined by the following procedures:

- Comparison of actual testing procedures to those specified in the QAPP.
- Comparison of analytical results of field duplicates to determine the spread in the analytical results.
- Examination of the analytical results of the QC blanks for evidence of contamination.

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