

**Environmental Security Technology Certification Program
(ESTCP)**

**In Situ Catalytic Groundwater Treatment Using Pd-Catalysts
and Horizontal Flow Treatment Wells
ER-0012**



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Final Report

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List of Acronyms and Abbreviations

AFB	Air Force Base
AFCEE	Air Force Center for Environmental Excellence
AFFTC	Air Force Flight Test Center
AFIT	Air Force Institute of Technology
AFOTEC	Air Force Operational Test and Evaluation Center
AFRPL	Air Force Rocket Propulsion Laboratory
AFRL	Air Force Research Laboratory
BEHIVS	Bioenhanced In-well Vapor Stripping
bgs	below ground surface
DBCP	1,2-dibromo-3-chloropropane
DCE	dichloroethylene
DHS	(California) Department of Health Services
DoD	Department of Defense
EE/CA	engineering evaluation / cost analysis (or cost assessment)
EICD	electro-conductivity detection
EPA	Environmental Protection Agency
ESTCP	Environmental Security Technology Certification Program
FSP	Field Sampling Plan
GC	gas chromatography
HASP	Health and Safety Plan
HazWopER	Hazardous Waste Operations and Emergency Response
HFTW	horizontal flow treatment well
ISACB	<i>In Situ</i> Aerobic Cometabolic Biodegradation
LLNL	Lawrence Livermore National Laboratory
MCL	maximum contaminant level (established under the Safe Drinking Water Act)
NASA	National Aeronautics and Space Administration
NAVFAC	Naval Facilities Engineering Command
NFESC	Naval Facilities Engineering Service Center
O&M	operations & maintenance
OSHA	Occupational Safety and Health Administration
PCE	perchloroethylene
Pd	palladium
PID	photo-ionization detection
PMC	Precious Metals Corporation, located in Sevierville, TN
POC	point of contact
PRB	permeable reactive barrier
RAB	Restoration Advisory Board
RPM	Remedial Program Manager (also Restoration Program Manager, Remedial Project Manager, and Restoration Project Manager)
QAPP	Quality Assurance Project Plan
SEM	scanning electron microscopy
SERDP	Strategic Environmental Research and Development Program
SPSS	Space Surveillance Squadron
TCE	trichloroethylene
VC	vinyl chloride
VOC	volatile organic compound
XPS	X-ray photo-electron spectroscopy

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Executive Summary

The U.S. Environmental Protection Agency (EPA) estimated in 1996 that approximately 70% of the 8,336 Department of Defense (DoD) sites requiring cleanup had contaminated groundwater, usually from chlorinated solvents such as trichloroethylene (TCE) and tetrachloroethylene (PCE). As a result, there is significant need for efficient treatment methods. Palladium (Pd) catalysis is a rapid destruction method that, in the presence of hydrogen gas, transforms many chlorinated ethylenes into ethane and some other halogenated volatile organic compounds (VOCs) into their respective hydrocarbon compounds. The dechlorination reactions for chlorinated ethylenes are complete and rapid and occur in water under ambient temperature, pH and pressure conditions. Hydrogen gas is used as the reducing agent, with residence times on the order of minutes. Catalytic contaminant destruction in a one-pass process has many potential advantages such as eliminating the secondary waste stream created by other processes that transfer contaminants to another medium (e.g. air or activated carbon). The technology is also effective in areas of high contaminant concentrations making it applicable to source control.

The objective of this project was to demonstrate the feasibility of catalytic destruction of chlorinated VOCs in groundwater using reactors containing palladium-coated beads that were operated in-situ within two previously established horizontal flow treatment wells (HFTWs). Unfortunately, deploying the reactors in situ proved an insurmountable challenge throughout the demonstration period and the reactors were operated above grade. Although installation of the reactors inside the treatment wells could be possible in a full scale application, it is not recommended due to complications associated with installing feed lines for backflushing and regenerating reactors coupled with high costs for removing reactors from the wells for maintenance, leak checks, etc.

The performance objectives of this study were to:

- (1) Demonstrate the efficacy of catalytic treatment for the destruction of chlorinated ethylenes in groundwater using palladium catalyst;
- (2) Optimize treatment efficiency; and,
- (3) Develop cost and performance data for full-scale application of the technology.

Collected data show process efficacy and a protocol for treating TCE contaminated groundwater was developed based on operational experience. On the basis of these developed parameters, the cost and performance for a dual-reactor system that treats a total of 4 gpm (2x 2 gpm) were evaluated. As part of the project, modeling was used to estimate the performance of a treatment system in conjunction with HFTWs. Modeling indicates that a series of HFTWs aligned perpendicular to the direction of groundwater regional flow could serve as an effective barrier to TCE migration.

Demonstration Results: Catalytic destruction of TCE in groundwater was demonstrated at Edwards AFB. The site was contaminated with 800 to 1,200 $\mu\text{g L}^{-1}$ TCE, which was the sole contaminant. A treatment methodology was developed to maintain catalyst activity and keep

treated water TCE concentrations at or below the maximum contaminant level (MCL) of $5 \mu\text{g L}^{-1}$ without byproduct formation. The treatment protocol entailed treating 2 gpm in a single catalyst column for 21 h (contact time approximately 1 min) followed by a 3 h bleach cycle to restore and maintain catalyst activity. The maintenance cycle consisted of bleaching of the catalyst for 1 h and flushing with hydrogen-containing groundwater for 2 h. After each maintenance cycle, TCE in the product water was at or below $1 \mu\text{g L}^{-1}$ corresponding to 99.9% removal. During a 21 h treatment cycle, effluent TCE concentrations increased slowly to approximately $10\text{-}15 \mu\text{g L}^{-1}$, corresponding to approximately 99% removal.

Daily bleaching maintained catalyst activity by preventing biological fouling with sulfidogenic bacteria (bacteria oxidizing hydrogen and reducing sulfate to hydrogen sulfide). Operational problems led to episodes of biological sulfide formation and severe catalyst poisoning marked by complete activity loss. Laboratory experiments and field observations demonstrated that the activity of the catalyst can be nearly completely recovered by treating the catalyst with bleach.

Based on data obtained in this demonstration, it is estimated that a capital investment of \$572,000 and annual O&M costs of \$72,000 (including monitoring & analysis) are sufficient to install and operate a treatment system that creates a barrier approximately 20 m wide in a plume of contaminated groundwater. This estimate applies to sites contaminated with chlorinated ethylenes (PCE, TCE, DCE isomers and vinyl chloride) with a relatively permeable aquifer, shallow water table and low gradient, similar to the Edwards AFB field site. This cost estimate is for a two-well system having a total flow of 2 gpm per treatment well or 4 gpm total. The system operates 87.5% of the time in a daily 21h:3h treatment:regeneration cycle and remediates a TCE concentration of $1000 \mu\text{g L}^{-1}$. The estimate is directly applicable to a full scale system and scalable to multiple sets of two wells. Sites with lower quality water would require more frequent bleaching whereas sites with cleaner (more aerobic) water are expected to require less frequent bleaching. A modification is proposed for continuous (100%) treatment by using two catalytic columns per well whereby one reactor is bleached and reactivated while the other treats the contaminated groundwater.

1. Introduction

1.1 Background

Groundwater contamination is a significant problem at thousands of Department of Defense (DoD) installations. The U.S. Environmental Protection Agency (EPA) estimated in 1996 that of 8,336 DoD sites needing cleanup, approximately 70% had contaminated groundwater [U.S. EPA, 1997]. The most common type of groundwater contamination is from volatile organic compounds (VOCs), found at approximately 75% of contaminated sites; the most common VOCs are chlorinated solvents such as trichloroethylene (TCE) and tetrachloroethylene (PCE). Based on EPA estimates, TCE and PCE contaminate groundwater at over 2,000 DoD installations. These contaminants are mobile and refractory in aerobic environments. There is significant need for efficient treatment methods because remediation of VOC sites using conventional pump-and-treat technology (i.e. activated carbon adsorption) is expensive and inefficient.

The objective of this project was to demonstrate the feasibility of catalytic destruction of chlorinated VOCs in contaminated groundwater. Catalytically destroying contaminants in a one-pass process has many advantages, chiefly that contaminants are completely destroyed instead of transferred to another medium (e.g. air or activated carbon), thus eliminating any secondary waste stream requiring further remediation. The technology is also applicable to high concentrations for control of contaminant sources where biological processes may be susceptible to toxic effects.

Laboratory and field studies have shown TCE, PCE and other halogenated VOCs can be destroyed in minutes by palladium catalysts contacted with dissolved hydrogen [Schreier and Reinhard, 1995; Siantar et al., 1996; Lowry and Reinhard, 1999; McNab et al., 2000]. In the process, chlorine atoms are replaced with hydrogen atoms forming products that are less toxic or benign in many cases. In the case of TCE, dechlorination is followed by saturation of the double bond, forming ethane and hydrochloric acid – the reaction is complete within minutes at ambient temperature. If hydrogen is present in excess, TCE dechlorination is complete and no chlorinated intermediates are formed. Palladium catalysts are commercially available, making the technology accessible to commercial users.

This report is organized as follows: The body of the report follows the required ESTCP format and describes the basics of the technology and demonstration design, summarizes performance and assessment of the technology, provides a the summary of the cost analysis (detailed in a separate report) and finally discusses implementation issues. APPENDIX A contains analytical methods supporting the experimental design, APPENDIX B is a description of relevant EPA methods, APPENDIX C is the quality assurance project plan, APPENDIX D is the health and safety plan, APPENDIX E is the design package for treatment system and APPENDIX F contains published reports related to the project.

1.2 Objectives of the Demonstration

The principal objectives of this study were to:

- (1) Demonstrate the efficacy of catalytic treatment for the destruction of chlorinated ethylenes in groundwater using palladium catalyst;
- (2) Optimize treatment efficiency; and,
- (3) Develop cost and performance data for full-scale application of the technology.

The study at Edwards AFB was close enough to full-scale that costs were scaled accordingly to represent full-scale application. In the initial proposal, the reactors were expected to be mounted below grade within the horizontal flow treatment wells (HFTWs), thus qualifying as an in situ technology. The test site was installed at the Edwards AFB site where the HFTW technology was tested previously in the context of biological treatment (McCarty et al. 1998). Experience gained during the execution of the project demonstrated that the best application of this technology at the Edwards AFB field site required a dual-column configuration with two reactors operating in tandem for each well, as explained below. Although a dual-column configuration in situ might be possible in principle, its realization was not feasible within the constraints of this pilot-scale demonstration. Once operational issues were resolved and the regeneration protocol was optimized, the catalyst reactor successfully reduced the TCE concentrations in the groundwater by 2-3 orders of magnitude (more than 99%) consistently and without significant loss of catalyst activity.

1.3 Regulatory Drivers

The primary health risk associated with TCE is cancer; the MCL adopted by EPA and the California Department of Health Services (DHS) is $5 \mu\text{g L}^{-1}$. California requires sites with contamination exceeding the MCL to provide treatment that lowers TCE concentrations to below $5 \mu\text{g L}^{-1}$. At the time that DHS adopted the MCL, it designated both packed tower aeration and granular activated carbon (GAC) as the best available technologies for TCE removal [California DHS, 2001]. As shown in this study, palladium catalyzed destruction is a potential cost effective strategy for meeting the MCL.

The cleanup of groundwater contamination at Site 19 is managed by the Environmental Management Office of Edwards AFB, and is overseen by the following regulatory agencies:

- U.S. Environmental Protection Agency, Region 9;
- California Department of Toxic Substances Control; and,
- Regional Water Quality Control Board, Lahontan Region (part of the California State Water Resources Control Board).

1.4 Stakeholder/End-User Issues

This demonstration evaluated the following potential stakeholder and end-user issues and came to the following conclusions:

- Palladium catalyzed destruction is a technology capable of treating groundwater contaminated with TCE, PCE, dichloroethylene isomers (DCE) and vinyl chloride at a wide range of concentrations;
- The operational conditions (bleaching duration and frequency, bleach concentration, catalyst regeneration) were optimized such that product water met treatment objectives and catalyst activity could be maintained for indefinite periods (years). Bleaching the reactor to prevent biological fouling and maintaining catalyst activity was the most critical operational issue. Catalyst cost and longevity were not important factors;
- Pilot-scale cost data were generated and can be used to estimate the cost of full-scale implementation, as provided in the Cost and Performance report. Full-scale implementation using the same method implemented at Edwards AFB is estimated at:
 - A one-time capital investment of approximately \$638,000; and,
 - Annual O&M costs of \$70,000.

The operating parameters at such a site would be close to those of this study: initial TCE concentration around $1000 \mu\text{g L}^{-1}$, hydrogen flow rate 250 mL min^{-1} , two parallel reactors (one reactor per well) with total flow 2 gpm, regeneration for 3 h daily. To cost for 24-hour operation, the cost estimate would need modification to include 2 reactors per well instead of the 1 per well used in this pilot study; and,
- Using the field experience of implementing a new and innovative technology, cost efficient and robust systems can be built and operated.

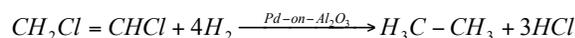
These conclusions are site specific and depend on water quality, hydrogeological conditions, and treatment and regulatory requirements and can be addressed by appropriate site specific pilot studies and hydrogeological investigations.

From a regulatory point of view, an important consideration was implementing the technology below surface to qualify as an in situ technology (as opposed to a pump-and-treat technology). To meet this objective, the design of the first system built and operated at the Lawrence Livermore National Laboratory (LLNL) [McNab et al., 2000] was followed, where the reactor was mounted below grade within a well. However, operating the reactor below the surface provided no technical benefits and many disadvantages; LLNL designed the second system for both above and below ground operation. For the Edwards AFB demonstration, the plan was to mount the reactors inside the treatment wells, above the sampling and treatment pumps, once testing and optimization was completed above ground. Due to technical challenges, in situ operation was not tested in this study.

2. Technology Description

2.1 Pd-Catalyzed Dehalogenation

Palladium (Pd) catalysts, in the presence of hydrogen gas, transform many chlorinated VOCs into their respective hydrocarbon compounds. To maximize the specific Pd surface area while minimizing the amount of metal used, a thin layer of Pd is supported on a porous support material such as porous gamma-alumina ($\gamma\text{-Al}_2\text{O}_3$). Pd catalyst transforms chlorinated ethylenes to ethane by replacing all chlorine atoms with hydrogen and hydrogenating the double bond. TCE, for example, reacts with 4 moles of hydrogen gas to form ethane and 3 moles of hydrochloric acid, as shown below:



This reaction is extremely rapid in water (nearly diffusion limited), even at ambient temperature, and proceeds completely to ethane [Lowry and Reinhard, 1999]. In the presence of excess hydrogen, no significant amounts of intermediates (e.g. vinyl chloride) are formed.

The formation of hydrochloric acid as a reaction product does not generally represent an obstacle for technology application to contaminated groundwater sites because the reactant TCE concentrations are generally low (less than 30 mg L^{-1}) and groundwater usually has some natural buffer capacity. If the contaminated groundwater contains high enough concentrations of chlorinated compounds (e.g. greater than 100 mg L^{-1} TCE) it is possible that enough hydrochloric acid would be formed to significantly alter the pH of the system, but this was not the case for the Edwards AFB groundwater site studied where TCE concentrations ranged from $800\text{-}1,200 \text{ }\mu\text{g L}^{-1}$.

The ability of Pd metal to catalyze dehalogenation reactions has been known for decades, but has only recently been applied to treatment of contaminated water. Previously, Pd-catalyzed hydrogenation or dehalogenation reactions were used primarily for synthesis of organic chemicals [Rylander, 1973]. Catalytic dehalogenation was applied to waste treatment in the 1980s, but it was either applied to organic waste streams [Kalnes and James, 1988] or required high temperatures or pressures to treat aqueous waste streams [Baker et al., 1989]. It was about a decade ago that Kovenklioglu et al. [1992] suggested Pd catalyst for ambient condition treatment of waste or groundwater contaminated by chlorinated hydrocarbons. Since that time, Reinhard and co-workers have investigated which contaminants are amenable to dechlorination or reduction via Pd catalysis, how fast the reactions occur and how to maintain catalyst activity over time [Schreier and Reinhard, 1995; Siantar et al., 1996; Lowry and Reinhard, 1999, 2000; Munakata, 2005; Davie and Reinhard, 2006].

A column reactor was designed for this demonstration in conjunction with HFTWs. The treatment system design was based on the subsurface reactor system that has been operated since 1999 at the LLNL [McNab et al., 2000]. That system relies on daily venting with air for

approximately 12 h to prevent growth of sulfidogenic bacteria and fouling – these operating conditions limit the overall efficiency of the system to about 50% [McNab et al., 2000]. To increase the operating time,, the Edwards AFB system was equipped with an automatic bleaching system to allow for more aggressive regeneration and fouling prevention protocols, relying on b
id Reinhard, 2000].

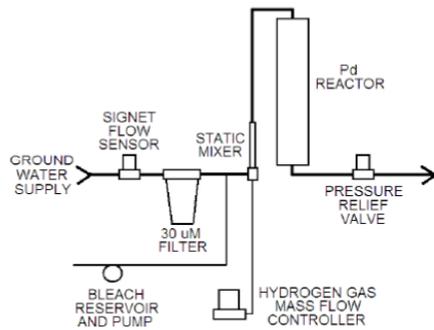


Figure 2-1: Simplified Schematic of Treatment System.

The system was built by a commercial vendor (Bigler and Associates, Lakewood, NJ) and delivered directly to the site. During the start-up phase, numerous components of the system had to be modified to meet the needs and conditions of the Edwards AFB site, significantly delaying operation and augmenting expenses, as discussed below.

2.1.1 Reactor Development

The treatment system was developed based on the designs of two previous systems and Stanford laboratory studies [Munakata, 2005]. The design packet developed by the LLNL team is provided in APPENDIX E. A reactor schematic and symbols legend are given in Figures 2-2 and 2-3, respectively. Five major considerations influenced the design:

- (1) The requirement to mount the reactors inside the existing treatment wells;
- (2) The need to operate the system at a remote location;
- (3) Budget and time constrains;
- (4) TCE effluent concentrations below the MCL ($5 \mu\text{g L}^{-1}$); and,
- (5) hydrogen safety concerns.

Requirement 1 was driven by regulatory standards (which have since been relaxed). To operate the reactor remotely, an internet-based system control was installed which added significant cost and was eventually deemed nonfunctional for this study. Financial and time constraints resulted in selecting the lowest bidder for construction and using limited factory support and testing onsite. Residence times and catalyst amounts per column were driven by meeting the MCL for TCE. Figure 2-1 is the simplified schematic of the final configuration used for the demonstration. Because of hydrogen safety concerns, a number of hydrogen sensors and safety features were installed, further augmenting total system cost and adding complexity. Figures 2-2 and 2-3 show the design schematic; details are given in APPENDIX E. Below, the major system elements and the associated control requirements are listed. The most important control requirements were:

- (1) Automatic system shutoff in the event of malfunctioning major system components, deviation from normal operating conditions, low bleach levels and dangerous levels of hydrogen gas;
- (2) In the event of system shut down, hydrogen flow to the reactors is discontinued and replaced with nitrogen gas (to prevent catalyst fouling); and,
- (3) Hydrogen is replaced with nitrogen during regeneration cycles. Safety interlocks could not be bypassed.

The major electronics requirements for the system were:

- (1) Groundwater pump (one for each treatment well)
 - a. 0-6.5 gpm flow rate
 - System shutdown on pump fault
 - Shutdown pumps on interlock trip (via relay contacts)
 - Operate flow rate via pump speed control
 - Manual on/off
 - Display on/off status
 - Set system pressure via pressure regulating valve
- (2) Flow meter (one per reactor)
 - a. 0-10 gpm Flow Rate Range; 4-20 mA Transmitter
 - Monitor / display flow rate at extraction
 - Monitor / display flow total (digital pulse count per gallon)
 - Interlock on flow rate high / low
- (3) Hollow fiber hydrogen contactor (one for each reactor)
 - a. Differential Pressure Transducer (2ea.)
 - -36.1 psi to 36.1 psi (4-20 mA)
 - Interlock on high / low DP
 - b. Hydrogen Flow Rate
 - Measure flow rate only (0-1000 sccm)
 - Interlock on high and low flow rates
 - c. 3-way valve (2 ea.)
 - Hydrogen approved solenoid valves
 - Automatic control
 - Switch to nitrogen on interlock trip
- (4) Switch to nitrogen during regeneration cycle

- a. Hydrogen flow bypass switch
 - Switch nitrogen to contactor
- (5) Personnel and hydrogen safety
 - a. Gas (H₂) LEL (Lowest Explosive Limit) monitor / transmitter (1 ea.)
 - b. Hydrogen detectors (3 ea.)
 - Monitor LEL level (0-100%; 4-20 mA) at well heads (2 detectors)
 - Monitor LEL level (0-100%; 4-20 mA) at hydrogen manifold (1 detector)
 - Interlock on 10% LEL at well heads and hydrogen manifold
 - Interlock on detector *I* monitor fail
 - c. System shutdown button
 - Interlock input
- (6) Miscellaneous interlocks
 - a. Gas pressure switches
 - Hydrogen supply (2 ea.) - interlock on low pressure
 - Nitrogen supply (2 ea.) - interlock on low pressure
 - Air supply (2 ea.) - interlock on low pressure
- (7) Regeneration and fouling control system
 - a. Bleach metering pump (2 ea.)
 - Automatic on/off control
 - Manual preset pumping volume
 - b. Bleach tank level switch
 - System shutdown on low level
- (8) System pressure (2 ea.)
 - a. Pressure transducer
 - Monitor (0-100 psi; 4-20 mA)
 - Interlock on high and low pressure setpoints
- (9) Control and remote operating system
 - a. PLC (programmable logic controller)
 - Digital / analog I/O
 - Remote communications link (modem)
 - Data processing
 - Control logic
 - b. Display panel
 - Operating parameters
 - Fault indicators
 - c. Interlocks control system
 - Any interlock fault shuts down entire system
 - Turn pumps off
 - Switch 3-way hydrogen valve to nitrogen supply

- Stop regeneration cycle
- Display first interlock fault input
- Display all subsequent interlock faults
- All interlock faults latch
- Manual reset pushbutton to clear interlock faults
- Interlocks bypass switch
 - Bypass process interlocks for start up
 - Safety interlocks never bypassed
 - One hour timeout enables all interlocks