ESTCP Executive Summary

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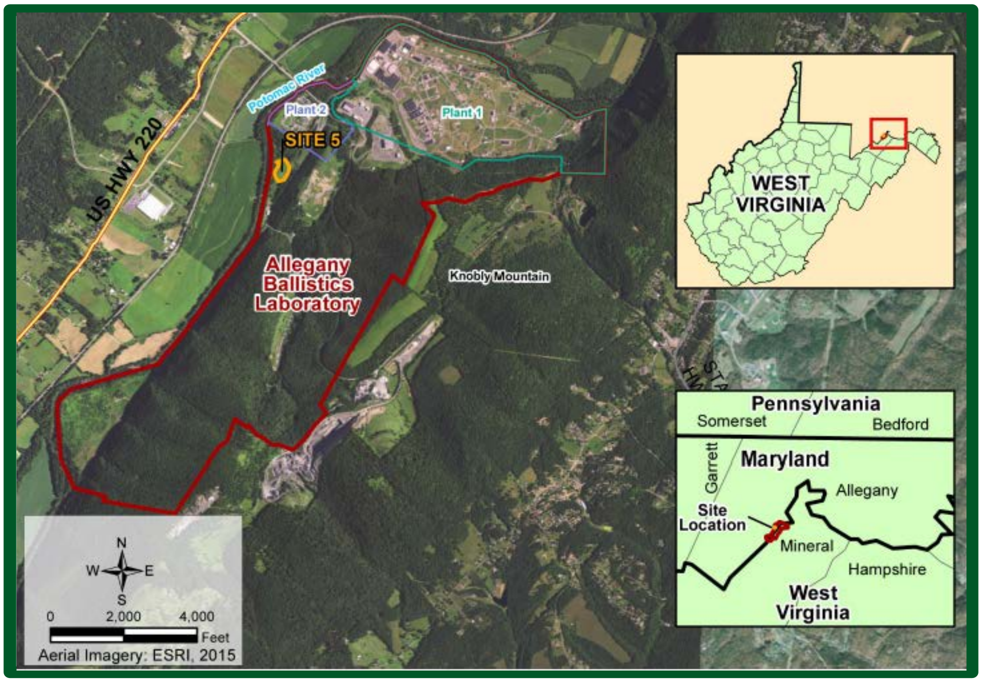

Analysis of Long-Term Performance of Zero-Valent Iron Applications

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ESTCP

ENVIRONMENTAL SECURITY
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14. ABSTRACT
This project involves the assessment of long-term performance of ZVI both as a source-zone treatment and as a barrier treatment for chlorinated volatile organic compounds (VOCs). This document details the field activities and data evaluation that were conducted in support of this project. The project approach consisted of a desktop review and field assessment. The field assessment was conducted at two selected sites. The first site was a Zero-valent Iron (ZVI) permeable reactive barrier (PRB) for plume control assessment at Allegany Ballistics Laboratory (ABL) Site 5. The other was at St. Louis Ordnance Plant Operable Unit 1 (OU1), where ZVI was introduced by soil-mixing in a source area.

15. SUBJECT TERMS
permeable reactive barrier, PRB, Allegany Ballistics Laboratory, ABL, St. Louis Ordnance Plant, zero-valent iron, ZVI, chlorinated volatile organic compounds, chlorinated VOCs, source-zone treatment, barrier treatment, groundwater

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EXECUTIVE SUMMARY

Project: ER-201589-PR

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1.0 INTRODUCTION

Environmental Security Technology Certification Program (ESTCP) Project Number ER-201589-PR, Analysis of Long-Term Performance of Zero-valent Iron (ZVI) Applications (the project), was completed to assess the long-term performance of ZVI both as a source-zone treatment and as a barrier treatment for chlorinated volatile organic compounds (VOCs). This executive summary briefly describes the results of the desktop study, field activities, and data evaluation that were conducted in support of this project.

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2.0 OBJECTIVES

Performance objectives for this project were to:

- Assess continued zero valent iron (ZVI) influence on geochemistry and contaminant chemistry at mature ZVI sites
- Determine the degree of mineralization of the ZVI at mature ZVI sites
- Determine the current degree of ZVI reactivity at mature ZVI sites
- Assess microbial community changes due to ZVI application
- Determine if ZVI application changed groundwater flow
- Develop pre- and post-treatment data requirements for a Remedial Project Manager (RPM) tool kit

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3.0 TECHNOLOGY DESCRIPTION

ZVI has been used for groundwater treatment since the 1990s through soil mixing, direct push injection, and permeable reactive barriers (PRBs). ZVI oxidation in groundwater releases hydrogen, lowers the oxidation/reduction potential (ORP), and typically increases pH. Resulting reactions cause destruction/degradation of chlorinated VOCs through β-elimination, hydrogenolysis, and dehalogenation.

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4.0 PERFORMANCE ASSESSMENT

This project was completed in two phases. The first consisted of a desk top review of nine sites. The following sites were evaluated during the desk top review:

- PRB Sites
  - Allegany Ballistics Laboratory (ABL) Site 5, Rocket Center, West Virginia
  - Boeing Michigan Aeronautical Research Center (BOMARC) OT-16, Joint Base McGuire-Dix-Lakehurst, New Hanover Township, New Jersey
- Injection Sites
  - St. Julien's Creek Annex (SJCA) Site 21, Chesapeake, Virginia
  - Naval Surface Warfare Center (NSWC) White Oak Site 13, White Oak, Maryland
  - Savannah Air National Guard (SANG) Base, Site 8, Garden City, Georgia
- Soil Mixing Sites
  - Arnold Air Force Base (AFB), Solid Waste Management Unit (SWMU) 16, Manchester, Tennessee
  - United States Army Corps of Engineers (USACE) St. Louis Ordnance Plant Operable Unit I, St. Louis, Missouri
  - Marine Corps Base (MCB) Camp Lejeune Site 89, Jacksonville, North Carolina
  - Naval Support Facility (NSF) Indian Head Site 17, Indian Head, Maryland

Desktop Review findings are summarized in Table ES-1.

Table ES-1. Summary of Desktop Review Findings

Desktop Review Objective	Findings
Evaluate trends in ORP, DO, and geochemical indicators of oxidation/reductive state from the baseline round of treatment to the most recent data available.	ORP values following treatment generally demonstrated effective treatment, with most sites achieving <-300 millivolts (mV) and many achieving <-400 mV. An extended duration of time before return to baseline conditions was observed, with six of the nine sites not returning to baseline ORP for at least 1000 days and three sites still at lower ORP than baseline at the time of this study (5-12 years post treatment). Similar reductions in DO were observed, with most post-treatment concentrations <0.5 mg/L. While increases in indicators of reducing conditions (e.g. sulfide, methane, etc.) were noted at a few sites, these data were generally not available.
Evaluate changes in inorganic concentrations following treatment where data were available.	Data for inorganics was not available for six of the nine sites evaluated. Where available, dissolved iron was generally noted to increase downgradient and within treatment areas. Dissolved arsenic increased in the one treatment area in which this constituent was evaluated (St. Julien's Creek Site 21).

Table ES-1. Summary of Desktop Review Findings (Continued)

Desktop Review Objective	Findings
Evaluate contaminant concentration trends (parent chemical and daughter products) in consideration of geochemical and redox state to determine longevity of ZVI efficacy and to evaluate the degree to which contaminant degradation/destruction is occurring through reductive β-elimination or through sequential hydrogenolysis.	Significant decreases in contaminant concentrations were noted in ZVI treatment areas, particularly at soil mixing sites. Four of the nine sites achieved concentration reductions of >99%. Generation of daughter products was observed at six of the nine sites evaluated. Two of the sites without daughter product formation had high dosing (Camp Lejeune Site 89 at 2% and Indian Head Site 17 at 1%). Four of the six sites with daughter product generation demonstrated subsequent reductions of these daughter products. Destructive processes at many sites appeared to be in part through β-elimination, particularly at sites with higher dosing; however, reductive dechlorination was also evident and important based on trend evaluations. Most sites reviewed did not show VOC rebound to baseline levels at the time the desktop review was completed, which was in most cases more than five years following treatment.
Compare designs and treatment outcomes of each implemented action and identify any best practices for future treatment.	Concentration reductions were highest at sites with higher ZVI:soil mass ratios. All sites with a ZVI:soil mass ratio of 1 or higher achieved >99% reductions. Soil mixing sites typically had the greatest reductions. At all sites evaluated, post-treatment monitoring generally focused on compliance, with little or no evaluation of changes in geochemistry, microbial populations, or site hydraulics. In many cases, baseline data were not optimal for trend evaluations or long-term performance assessment (for example, some sites had no baseline hydraulic or geochemical data in the treatment area). The desktop review supports a minimum dosing of 0.4 ZVI:soil mass ratio, soil mixing when feasible, and comprehensive collection of baseline data to ensure performance monitoring can be completed, if necessary.
Review groundwater flow data to determine the potential for preferential flow around treated areas due to reduced hydraulic conductivity and "plugging" from mineral precipitation in the pore spaces of the treatment zones.	Obvious changes in flow direction or characteristics were not evident based on the limited data available for the desk top study.
Evaluate the presence or absence of a "clean front" on the downgradient side of Permeable Reactive Barrier (PRB) Sites.	At the McGuire PRB Site, no clean front was noted; however, a 33% reduction in concentrations was noted downgradient of the PRB. Average concentration reductions were more significant (70%) at the ABL site, but the monitoring well network was not optimal for assessing a clean front because contaminants were downgradient of the wall at the time of the installation and there were no wells close to the wall.
Identify two sites (one PRB site and one injection site) to be carried forward into the field portion of the project.	ABL Site 5 was identified as the preferred PRB site for field study because the remedy for this site was the more effective of the PRB sites and the trenched wall configuration was ideal for collection of remaining iron. St. Louis Ordnance Plant OU1 was selected as the preferred source area treatment site because the remedy was highly effective (average concentration reduction of 99.8%) and no clay was mixed with the ZVI, making it possible to attribute all reductions in concentrations to ZVI treatment rather than sorption.

Notes:

ABL	Allegany Ballistics Laboratory		
DO	dissolved oxygen	ORP	oxidation/reduction potential
mg/L	milligrams/Liter	PRB	permeable reactive barrier
mV	millivolts	ZVI	zero-valent iron

The field assessment phase of the project was focused on collecting and analyzing additional field measurements to assess long-term performance because most data available during the desk top review were focused on compliance. Field data were collected at ABL Site 5, located in Rocket Center, West Virginia, and the former St. Louis Ordnance Plant OU1 located in St. Louis, Missouri. The ZVI PRB at ABL was installed in the year 2006. Field efforts at ABL Site 5 included direct-push sampling of angled cores collected through the ZVI PRB, as well as installation of monitoring wells in two transects across the PRB (upgradient to downgradient) and in locations cross-gradient of the PRB. Field efforts at the former St. Louis Ordnance Plant OU1, a soil mixing ZVI application, included collection of ZVI/soil cores within the mixing area and upgradient and downgradient of the mixing area, as well as installation of six new temporary wells: two within the mixing area, one upgradient and one downgradient of the mixing area, and two cross-gradient of the mixing area. The soil-mixing ZVI application at this site was conducted in the year 2012. The objective of these additional field measures was to better evaluate both chemical and hydrogeologic performance of the ZVI applications.

Groundwater samples were collected from the newly installed wells and select existing wells at each site for analysis of VOCs, total and dissolved metals, silica, strontium, sulfide, nitrate, nitrite, ammonia, total organic carbon, hardness, alkalinity, methane, ethane, ethene, acetylene, and the following anions: sulfide, chloride, phosphate, and fluoride. These analyses were completed by Microbac Laboratories. Additionally, microbial samples were collected by pumping water through laboratory-provided biofilters and sending the filters and volume pumped to Microbial Insights of Knoxville, Tennessee for next generation sequencing (NGS) and QuantArray-Chlor analysis. ZVI cores were sent to the United States Environmental Protection Agency (USEPA) Risk Management Research Laboratory for mineralogical analysis. As a separate supplement, duplicate ZVI cores and up- and downgradient reference soil cores were sent for reactivity analysis to Oregon Health and Science University (OHSU), which is conducting its own ESTCP-funded study.

Notable changes in chemistry were observed in the aquifer downgradient of the PRB at ABL and inside the treatment area at St. Louis. Decreases in ORP and dissolved oxygen (DO), as well as increases in pH were observed downgradient of the ZVI application at ABL. However, it should be noted that in one transect of the ABL PRB, lowest ORP values were observed in the well furthest downgradient of the PRB, indicating PRB effectiveness has likely diminished over time. Similar trends were observed in pH data for the same transect. In the soil mixing area at the St. Louis Site, the ORP has rebounded from below -400 mV following treatment to -273 mV in the one well within the treatment area prior to this study. However, ORP remained below -400 mV in one of the temporary wells in the treatment area installed to support this investigation, indicating possible continued reactivity of ZVI. DO and pH were also indicative of continued activity of the ZVI at St. Louis.

At ABL, decreases in calcium, magnesium, and strontium were observed downgradient of the PRB, indicating that the PRB may be continuing to act as a sink for these metals. At the St. Louis site, concentrations of calcium, magnesium, barium, and strontium were highest in the upgradient portion of the mixing area.

Non-detect concentrations of VOCs immediately downgradient of the PRB at ABL indicated that a “clean front” is emerging in the eastern transect across the wall. Concentrations of VOCs in the new wells installed by this project at the St. Louis site were slightly higher than levels previously detected in existing wells, both within and outside of the mixing area, but did not exceed the previously established clean-up goal for the site. The permanent monitoring well in the central portion of the treatment area continues to show non-detect concentrations or very low detections (less than maximum contaminant levels [MCLs] for all VOCs in comparison to the highest concentration of 36,100 µg/L pretreatment).

Results of mineralogical analysis indicated weathering of ZVI at both sites. Electron micrographs and x-ray mapping of the iron from the ABL site indicated a mottled appearance indicating corrosion. Similarly, a significant reduction in iron particle size was noted at the St. Louis site in comparison to an iron reference sample provided by the iron supplier for that site. X-ray diffraction (XRD) results for both sites identified minimal elemental iron in cores analyzed by the lab; however, these cores were from a limited number of locations and may not be representative of conditions throughout either treatment area. Energy dispersive line scans and x-ray absorption near edge structure (XANES) data from the same core samples were also indicative of minimal elemental iron remaining in the cores analyzed at the St. Louis site. At ABL, the presence of iron oxides was noted (magnetite and hematite). At St. Louis, the iron identified was primarily magnetite and goethite. Acid-volatile sulfur (AVS) data from ABL indicated the presence of iron sulfide. While overall results indicated the minimal elemental iron in the limited sample set collected, the potential for secondary reactivity through minerals such as magnetite was identified at both sites. Additionally, geochemistry described in the sections that follow indicates reducing conditions indicative of presence of ZVI that was not observed during microscopy analysis of cores. Build-up of precipitates on the iron and quartz components of the wall was observed through Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS), but was minor and did not result in excessive cementation of grains that would likely result in hydraulic plugging.

Microbial data collected at the ABL PRB site did not indicate the presence of high numbers of microbes or genes commonly associated with reductive dechlorination of VOCs. However, NGS data do indicate significant changes in overall microbial populations resulting from the PRB, with *Sulfurimonas*, a sulfur oxidizing bacterium, dominant on the downgradient side of the wall, possibly taking advantage of reduced sulfur exiting the wall. *Firmicutes*, which are known acetylenotrophs, were also noted downgradient of the wall, which may explain the lack of detections of acetylene at the site if generation of acetylene is outpaced by consumption. At the former St. Louis Ordnance Plant, populations of reductive dechlorinators are higher within the treated area than outside. While *Dehalococcoides* species (sp.) and its functional genes known to be involved in complete dechlorination of VC were not present in significant quantities, other reductive dechlorinators (e.g., *Dehalogenimonas*) appear to be present along with aerobic metabolizers and cometabolizers of VC (both ethenotrophs and methanotrophs). It is unclear from the data collected whether these typically-aerobic bacteria are active and present in aerobic microenvironments within the mixing area, whether they are tolerant to anaerobic conditions (as has been demonstrated to be possible for some ethenotrophs [Liang, 2017]) or whether they may be dormant. NGS data were also indicative of changes in the microbial population, with higher population percentages of the phylum *Firmicutes* and the genera *Alkaliphilus*, *Sulfuricurvum*, and *Methylotenera* within the treatment area relative to outside, supporting the continued changes in geochemistry created by the ZVI. Similar to the ABL site, the presence of *Firmicutes*, a known acetylenotroph, may explain the lack of detections of acetylene at the former St. Louis Ordnance Plant.

Water level data were evaluated to determine impacts on flow characteristics at both sites. Addition of new monitoring wells within and closer to the ZVI enabled a better evaluation of the hydrology of the applications. Additionally, slug testing data were evaluated for the St. Louis site only. For the ABL site, flow direction observed was slightly different from previous evaluations, possibly due to seasonal variability or as a result of the more extensive monitoring well network added. No mounding was observed, and flow direction did not indicate the potential for contaminants to migrate around the PRB. Consistent with the SEM and EDS data, groundwater flow data at ABL are not indicative of a build-up of precipitates that is impacting the hydrologic characteristics of the PRB. At the St. Louis site, measured hydraulic conductivity from slug tests did not indicate differences between the mixing area and outside-of-the-mixing area.

The findings from the field assessment, in the context of the overall project Performance Objectives, can be summarized as follows:

- **Assess continued zero valent iron (ZVI) influence on geochemistry and contaminant chemistry at mature ZVI sites:** There is evidence in the soil-mixing application that strongly reducing conditions (-400 millivolt [mV] or lower) conducive to β-elimination reactions were generated in some parts of the treatment zone for at least a year following application, leading to considerable abiotic degradation of tetrachloroethene (perchloroethene [PCE]) and trichloroethene (TCE) without much generation of cis-dichloroethene (cis-1,2 DCE) and vinyl chloride (VC). Five years after application, these strongly reducing conditions persist in some parts of the treatment zone, but not in others, where weaker reducing conditions conducive to biodegradation still prevail. At a minimum, reducing conditions conducive to sequential biodegradation of chlorinated VOCs were generated by the ZVI at both sites. These reducing conditions continue to prevail at the PRB and soil-mixing sites after application for eleven and five years, respectively. Minerals formed on the ZVI at both sites may contribute to additional reducing (abiotic) reactions that are weaker than those with fresh ZVI, but may nevertheless provide additional protection in the longer term.
- **Determine the degree of mineralization of the ZVI at mature ZVI sites:** Data from the ABL site indicated ZVI weathering with some passivation resulting from precipitation of coatings and transformation to less reactive iron minerals. At the St. Louis site, evidence suggested that no identifiable ZVI remained in cores from the mixing area. Iron identified was primarily magnetite and goethite. Overall, results indicate weathering of the ZVI.
- **Determine the current degree of ZVI reactivity at mature ZVI sites:** There is evidence from the new wells installed by this project closer to the PRB that the ZVI is becoming progressively weaker over the years. Eleven years after the PRB application, more strongly reducing conditions are evident progressively further downgradient of the PRB, thus indicating that ZVI performance has peaked sometime in the past. The PRB has been progressively less strongly reducing, no longer conducive to β-elimination reactions, but possibly conducive to some continued biodegradation.
- **Assess microbial community changes due to ZVI application:** Evidence for microbial community shifts conducive to partial reductive dechlorination was site specific. Selection for microbiological dechlorination metabolism was observed at the St. Louis site but not at ABL.

- **Determine if ZVI application changed groundwater flow:** The hydrology at both sites indicates that groundwater continues to flow relatively unhindered through the ZVI PRB and soil-mixed area in the two applications.

Overall, the desktop review and the more detailed field study at the two sites indicates that ZVI performed best at shorter term mass removal, such as the source treatment at the soil-mixing site. In a longer-term plume control application, strongly reducing conditions conducive to continued abiotic (β-elimination) reactions may not last for the duration of the plume. Weaker reducing conditions conducive to biodegradation reactions and mineral formation implicated in weaker abiotic reactions may help maintain some degree of continued plume control. Their usefulness will depend in the longer term on the continued strength of the upgradient plume and the ability of the aquifer (and native microbial populations) to take the sequential degradation reactions to completion (to ethene and ethane, while minimizing any accumulation of cis-1,2 DCE and VC).

A tool kit of recommendations was developed for future assessment of ZVI remedies during the planning, execution, and performance monitoring phases and is presented in Table ES-2.

Table ES-2. Recommended Best Practices

Category	Observation	Recommended Best Practice
Pre-Remedy Selection	At sites with high DO and ORP, natural reductant demand may more-rapidly deplete ZVI, impacting remedy effectiveness	ORP and DO should be carefully considered prior to selection of ZVI remedies. In cases where DO and ORP are very high, other remedies more compatible with oxidizing conditions may be more effective. Currently, natural oxidant demand testing is common when assessing in situ chemical oxidation (ISCO) remedies, but the natural reductant demand of aquifers is not often assessed prior to implementing chemical reduction remedies.
	At PRB sites, contamination is often observed downgradient of the wall following installation. Additionally, flow direction may be seasonably variable resulting in the PRB not remaining perpendicular to groundwater flow at times.	When feasible, PRB design should be completed after installation and sampling of monitoring wells downgradient, upgradient, and cross-gradient of the proposed PRB. This will allow for optimization of wall position.
	At sites where contaminant concentrations were delineated using DPT, groundwater geochemistry and field parameter data were often not available for the period prior to remedy implementation in the treated area.	Collect some baseline geochemistry and field data in the highest concentration areas to assist in the evaluation of treatment effectiveness once iron treatment is employed.

Table ES-2. Recommended Best Practices (Continued)

Category	Observation	Recommended Best Practice
Remedy Implementation, Performance Monitoring, and Optimization	Lack of pre-implementation geochemical data in the immediate downgradient vicinity of an PRB installed within the groundwater contaminant plume limits the assessing the PRB's performance due to effect of desorption/diffusion of contaminants.	Collect two rounds of geochemical data prior (within a year) to installation of ZVI application in the area 5-15 feet downgradient of the planned application. Plan on a site visit by the Remedial Design team 90-95 percent submission to layout ZVI application align/area as closely as possible so that permanent or temporary groundwater monitoring wells can be installed.
	Effectiveness is highly dose-related (ZVI to soil ratio) with mixing areas at which doses were >1% generally achieving the best results	While doses of 0.5% may be sufficient at some sites, designs of >1% are generally effective.
	While aquifer ORP was often consistent with conditions favorable for dechlorinating microbes, such as *Dehalococcoides* sp., these microbes were not present in abundant concentrations and/or with ideal functional genes downgradient of or within treatment areas at either field study site, possibly as a result of generally low organic carbon concentrations or sub-optimal native microbial populations.	If a treatment train is desired in which anaerobic conditions created by ZVI are intended to facilitate reductive dechlorination downgradient of the ZVI treatment area, addition of organic carbon or bioaugmentation amendments may be necessary.
	At the St. Louis site, ZVI was found to have converted to magnetite over time in the small number of samples evaluated. At the ABL PRB site, iron was present primarily in the form of magnetite and hematite. Iron particles at the upgradient interface exhibited some mineral precipitates on their surfaces, primarily calcium carbonate and iron oxide. Minimal ZVI was observed in the few samples collected from St. Louis 5 years after treatment, though sample cores were not likely representative of the entire mixing area and the remaining magnetite still facilitated reductive activity based on reactivity analysis. Geochemical and microbial parameters at both sites were supportive of continued activity of the iron over time. While some signs of ZVI depletion were evident based on reactivity testing and mineralology testing of the limited sample set, geochemistry indicated highly reducing conditions, indicating the potential for more ZVI to be present in areas not sampled.	Because magnetite may still facilitate abiotic degradation of chlorinated VOCs, conversion of ZVI to magnetite is not entirely inconsistent with continued treatment. Additionally, build-up of precipitates which would inhibit reactivity at the ABL site was more common in portions of the wall at the upgradient interface, likely allowing for continued reactivity within the wall. However, monitoring of reactivity using redox indicators, such as resazurin, or batch reactors may be useful in determining the need for enhancements to mature iron remedies. Additionally, if microscopic analysis is completed, a larger sample set may be necessary to adequately assess the presence/absence of remaining ZVI.

Notes:

DO dissolved oxygen
DPT direct push technology
ISCO in situ chemical oxidation
ORP oxidation/reduction potential
PRB permeable reactive barrier
VOC volatile organic compound
ZVI zero-valent iron

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5.0 COST ASSESSMENT

The intent of this project was to assess long-term effectiveness of ZVI remedies. Implementation costs associated with ZVI remedies have been evaluated as part of the studies listed below:

- ESTCP. 2010. *Cost and Performance Report Emulsified Zero-valent Iron Nano-scale Iron Treatment of Chlorinated Solvent DNAPL Source Areas (ER-200431)*. September.
- NAVFAC. 2012. *Permeable Reactive Barrier Cost and Performance Report*. March.
- NAVFAC. 2008. *Cost and Performance Report for a Zero Valent Iron Treatability Study at Naval Air Station, North Island*. July.

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6.0 IMPLEMENTATION ISSUES

Because the scope of this project involved evaluation of remedies that have already been implemented, no new information on implementability was collected. However, a thorough review of implementation of ZVI remedies is available in the following documents:

- Interstate Technology and Regulatory Council (ITRC). 2005. *Permeable Reactive Barriers: Lessons Learned/New Directions.* February.
- ITRC. 2011. *Permeable Reactive Barrier: Technology Update*. June.
- Powell, R. M., P. D. Powell, and R. W. Puls. 2002. *Economic Analysis of the Implementation of Permeable Reactive Barriers for Remediation of Contaminated Groundwater.* EPA/600/R-02/034. U.S. Environmental Protection Agency.

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