



ANNUAL REPORT

In Situ Chemical Oxidation Initiative

July 2004

Report Documentation Page				Form Approved OMB No. 0704-0188	
Public reporting burden for the collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to a penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.					
1. REPORT DATE 01 JUL 2004		2. REPORT TYPE N/A		3. DATES COVERED -	
4. TITLE AND SUBTITLE In Situ Chemical Oxidation Initiative Annual Report				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S)				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Environmental Security Technology Certification Program (ESTCP)				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release, distribution unlimited					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UU	18. NUMBER OF PAGES 12	19a. NAME OF RESPONSIBLE PERSON
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified			

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1. INTRODUCTION

The Strategic Environmental Research and Development Program ([SERDP](#)) and Environmental Security Technology Certification Program ([ESTCP](#)) are designed to develop and transition innovative research and technology to help the Department of Defense (DoD) perform its mission in several environmental [thrust areas](#). Environmental cleanup is one of these thrust areas. While DoD facilities may have several contaminants, chlorinated solvents are by far the most prevalent. These compounds, which collectively may be categorized as chlorinated aliphatic hydrocarbons (CAHs), continue to be difficult to remediate, despite several years of research and development. Cleanup is particularly difficult at sites containing CAH as dense nonaqueous-phase liquid (DNAPL) where the DNAPL serves as a continuing long-term source of dissolved-phase groundwater contamination. In Situ Chemical Oxidation (ISCO) has been rapidly adopted as a remediation technology for CAHs, and more recently for DNAPL source zones.

1.1 Background

SERDP and ESTCP are currently funding a number of [projects](#) in the area of ISCO development. A Technical Review Panel of experts from academia and the consulting industry provides SERDP/ESTCP project management with objective professional evaluations of progress made on these projects, identifies knowledge gaps in ISCO research and development, and recommends potential areas of funding to SERDP/ESTCP project management. This technical oversight format is unique in the field of cleanup technology research and development and ensures continuity and cross-fertilization in this focused effort to elucidate the benefits of ISCO remediation. Technical Panel members include:

Dr. Andrea Leeson, SERDP/ESTCP
Dr. Marvin Unger, The RETEC Group Inc.
Mr. Scott Dockum, HydroGeologic, Inc.
Dr. Dick Brown, Environmental Resources Management
Dr. Ian Osgerby, USACOE New England District
Dr. Bob Norris, Brown and Caldwell
Mr. Mike Marley, Xpert Design and Diagnostics, LLC

This report reviews the technical basis for funding priorities, highlights the scope and objectives of the individual projects, and summarizes their progress in advancing the understanding of key ISCO issues. The report is updated annually and is intended to meet SERDP's mandate to [transfer](#) science and technology emerging from these projects to regulators, cleanup specialists and the regulated community. A number of additional resources on the subject of ISCO remediation are available [elsewhere](#) on this website.

1.2 Understanding ISCO

Chlorinated solvents are the most prevalent organic contaminants in groundwater. Chlorinated aliphatic hydrocarbons (CAHs), such as trichloroethylene and perchloroethylene, are found at approximately 80% of all Superfund sites with groundwater contamination and more than 3000 Department of Defense (DoD) sites in the United States (*1*). The life-cycle costs to clean up these sites are uncertain, but are likely to exceed several billions of dollars nationally. DoD alone could spend more than \$100 million annually for hydraulic containment at these sites, such as using pump-and-treat technologies, and estimates of life-cycle costs exceed \$2 billion

ISCO involves injection of strong oxidants into the contaminated subsurface, in some cases with other chemicals that function as catalysts. The oxidants chemically break down CAHs upon contact to inert materials such as carbon dioxide, chloride and water. Chloroethenes are particularly susceptible to chemical oxidation. Several oxidants have been tried, but most commercial applications have used either hydrogen peroxide or potassium permanganate in the saturated zone and either hydrogen peroxide or ozone for the vadose zone.

The potential benefits from ISCO include in situ contaminant destruction, relatively low cost, reliability, simplicity (as compared to in situ biological treatment) and rapid treatment. Like any in situ technology, site-specific constraints must be considered. Oxidation is dependent on achieving adequate contact between oxidants and contaminants, and subsurface heterogeneities, preferential flow paths, and poor mixing in the subsurface can result in extensive pockets of untreated contaminants. Further, the reagents can be consumed by other oxidizable substrates (e.g., natural organic compounds or dissolved iron), limiting the efficiency of ISO treatment.

In situ oxidation has only been commercially practiced for the last 9-10 years. As a result, the technology is rapidly evolving and the state of the art has advanced considerably over time. The limitations of the technology are becoming better understood, and engineering approaches to overcoming some of these limitations have been developed. Although the chemistry involved is relatively simple, the technology is not a simple one to implement. The subsurface environment can be difficult to control, and it can be difficult to get adequate distribution of the oxidants within the subsurface. Often, site-specific data is needed that may not be available from typical site characterization investigations. There have been significant improvements the ability to distribute oxidants within the subsurface and an improved understanding of the site-specific data needs.

1.3 Research and Development Priorities

1.3.1 Improved Understanding of ISCO

The degree of success of ISCO demonstrations has varied significantly as a result of the limited understanding of the chemical reaction mechanisms and kinetics, the interactions between the oxidant, contaminants, and the aquifer matrix, the effects of secondary water quality constituents on oxidation reactions and by-product generation, and design constraints for successful application.

1.3.2 Reaction Kinetics

Few detailed studies have been reported on the kinetics and mechanisms by which permanganate oxidizes key groundwater contaminants, with available literature focusing predominantly on chlorinated ethenes. The literature on oxidation of organics by Fenton's reagent is larger; however, the scopes of most of these studies are narrow with respect to the number and type of contaminants considered. The lack of a comprehensive perspective on the chemistry of ISCO technologies is evident from the numerous inconsistencies in how oxidation by permanganate or Fenton's reagent are treated by most environmental chemists and engineers. For example, the strength of both oxidants is frequently equated with their very positive oxidation potentials by ISCO advocates, even though it is unusual for either oxidant to react by electron transfer. For permanganate, it is now realized that the current mechanistic model is incomplete; and, with respect to groundwater applications of Fenton's reagent, similar inconsistencies exist. Despite these uncertainties in the mechanism of contaminant degradation by permanganate and Fenton's reagent, complex and detailed kinetic models have been described for these oxidants. The development and application of these models has advanced our understanding of advanced oxidation processes; however, these models have rarely been applied in the field by practitioners of this technology. A research and development goal is to bridge the gap between the most detailed kinetic models and the highly simplified kinetic models used in engineering design.

1.3.3 Natural Oxidant Demand/Oxidant Mobility

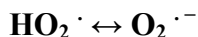
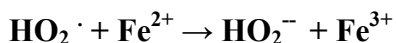
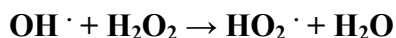
While an understanding of the reactivity of various contaminants with either permanganate or Fenton's reagent provides critical data for the appropriate application of these oxidants, the reactivity of the oxidant with naturally-occurring reductants and catalysts associated with the aquifer matrix imposes a competing oxidant demand that can impact the mobility of the injected oxidant. Characterization of the oxidant-related properties of this natural oxidant demand (NOD) is required for the design and evaluation of ISCO systems. If the impact of the NOD is not taken into consideration, the concentration of the oxidant may be depleted by the NOD to such a degree that the effect of the oxidant on the target contaminants is minimal. The presence of natural organic matter (NOM) and reduced mineral species (e.g., iron, manganese, and sulfur) provides a natural oxidation capacity that results in consumption of the injected oxidant until the ultimate oxidation capacity is satisfied or the oxidant is depleted. In addition to the NOD, a number of reactions also occur in the subsurface that may reduce the stability of the injected oxidants including formation of MnO_2 that can decompose permanganate, formation of metal oxides that can increase the rate of H_2O_2 decomposition, heat generated from exothermic reactions, carbonate dissolution resulting in CO_2 generation, and O_2 produced by H_2O_2 decomposition. Currently, there is no established methodology to determine the NOD of the aquifer matrix and only limited NOD data for either permanganate or Fenton's reagent is available in the literature. Therefore, a comprehensive understanding oxidant mobility and stability in the subsurface during ISCO treatment is required to assess the site specific applicability of ISCO, design oxidant delivery systems, and evaluate system performance.

1.3.4 Long-Term ISCO Impacts on Site Conditions

Despite the growing use of ISCO technologies at DoD sites, there is only a limited understanding of the effects of ISCO on long-term changes in groundwater quality, and the duration of these impacts is generally unknown. The anticipated impacts of ISCO will be oxidant-dependent, but the duration of their impacts may be broadly characterized by the types of reaction products that result. In the case of aqueous products that are essentially conservative (e.g., chloride), the impact on water quality may reasonably be expected to be transient as the ISCO treatment zone is flushed with background groundwater following ISCO application. Conversely, reaction products that can participate in reactions with ambient groundwater, such as trapped gas (e.g. O₂) or solid phases (e.g., MnO₂) will provide a reservoir of reaction product that may interact with groundwater for long periods of time following ISCO treatment. No data is available describing the long-term impacts of gas generation by Fenton's reagent on aquifer conductivity; and, the formation of MnO₂ may also reduce media porosity. Whether these impacts are likely to disappear over time and comply with regulatory criteria, or whether effects will be indefinite, is uncertain.

1.3.5 Alternative Mechanisms in Fenton-Like Reactions

All transformations by Fenton's reagent have traditionally been attributed to a hydroxyl radical mechanism. However, it is now believed that at least one other reactive species is generated by the catalyzed decomposition of hydrogen peroxide. Non-hydroxyl radical transient oxygen species are generated in increasingly high concentrations by propagation reactions as the hydrogen peroxide concentration is increased in a Fenton's system:



Superoxide anion (O₂ ·⁻) is a reductant and a weak nucleophile. Hydroperoxide (HO₂·) is a reductant and a strong nucleophile. Recent results show that the production of these species is dependent not only on hydrogen peroxide concentration, but also on the nature of the catalyst used. While some Fenton's catalysts generate hydroxyl radicals, some generate reductants, and some generate both hydroxyl radicals and reductants. Superoxide, (or hydroperoxide) may not only be responsible for the enhanced treatment of sorbed contaminants, but also for the enhanced destruction of DNAPLs. It is hypothesized that superoxide diffuses through groundwater to DNAPLs where it then diffuses into the DNAPL and reacts to destroy it from within. Some trace minerals may provide the key to catalyzing the species response for this mechanism, which may explain why modified Fenton's ISCO has been successful at some sites, but not at others.

The combination of hydroxyl radicals, superoxide, and hydroperoxide anions can potentially desorb contaminants and disrupt DNAPLs; furthermore, they can oxidize reduced compounds and reduce oxidized compounds, increasing the likelihood of mineralization of recalcitrant contaminants. Furthermore, the natural mineralogy of the subsurface may be critical to the effective catalysis of hydrogen peroxide, requiring sufficient reductant-producing trace minerals for the process to be effective. A better

understanding of subsurface geochemistry in conjunction with modified Fenton's reactions that generate all three transient oxygen species have the potential to provide a universal treatment matrix for ISCO. Thus, an understanding of the most effective mechanisms for generating these species in optimized proportions is essential for the successful full-scale implementation of modified Fenton's reagent.

1.3.6 Coupling ISCO with Other In Situ Treatment Technologies

The coupling of compatible in situ treatment technologies is a logical remediation approach particularly for more heavily contaminated source areas and plumes as well as DNAPL residuals. For source areas with high concentrations and DNAPL residuals, if there is adequate permeability and limited heterogeneity, high mass removal efficiencies can be achieved by flushing with surfactants/cosolvents, by thermal treatment, or by air sparging. Unfortunately, while these methods can achieve high mass recovery efficiencies of DNAPLs (e.g., 95% or more), there are still appreciable levels of residual contaminants in the subsurface (e.g., 1,000 mg/kg range) which can continue to sustain concentrations above risk-based action levels. In addition, the residuals may have an altered mobility due to treatment-induced changes in the contaminant and/or the subsurface and thus the residuals may be of great concern. Coupling a treatment strategy involving first mass recovery followed by a lower-level oxidant flush could cost effectively reduce the residual mass and enable achievement of clean-up goals such as maximum contaminant levels (MCLs) in groundwater. However this approach has not been explored in any detail as of yet.

Another coupling strategy involves combining ISCO with post-ISCO bioremediation. At some sites, one or more of the organics in a mixture are recalcitrant to biodegradation due to their original concentrations or chemical forms. Use of a pretreatment step involving ISCO could enhance subsequent biodegradation. However, strong chemical oxidants are known to be potentially toxic to microorganisms. Precipitates such as iron hydroxides and manganese oxides formed during chemical oxidation have also raised treatment concerns. Despite these concerns, numerous studies have verified the capacity of chemical pretreatment with oxidants such as peroxide, Fenton's reagent, and ozone to enhance subsequent biodegradation of organics in water or wastewater. Nonetheless, few studies have carefully investigated the effect of chemical oxidation on soil microbial activities or communities.

2. SERDP ISCO PROJECTS

The projects included in this initiative consist of three ongoing projects started in 2002. Progress on these projects is summarized below.

2.1 Project CU-1288: Improved Understanding of Fenton-Like Reactions for in Situ Remediation of Contaminated Groundwater Including Treatment of Sorbed Contaminants and Destruction of DNAPLs (Rick Watts, Washington State University)

Key Tasks/Results

One of the primary limitations of the use of modified Fenton's reagent is the high rate of hydrogen peroxide decomposition catalyzed by naturally-occurring minerals present in the subsurface. Phosphate stabilizers are ineffective in controlling hydrogen peroxide decomposition that is promoted by metal oxides and oxyhydroxides. If hydrogen peroxide stability is to be achieved in soils and the subsurface during Fenton's ISCO, the minerals that control its decomposition must be elucidated. The purpose of this research was to investigate hydrogen peroxide decomposition rates catalyzed by naturally-occurring minerals, and to confirm the minerals responsible by evaluating hydrogen peroxide decomposition rates in natural subsurface solids.

Samples of 14 naturally-occurring subsurface minerals and eight different subsurface solids collected from sites through North America were investigated for their potential to decompose hydrogen peroxide. Hydrogen peroxide residuals as a function of time were plotted for each of the 14 minerals. All of the trace minerals (e.g., anatase, bauxite, etc.), with the exception of cuprite, decomposed hydrogen peroxide at rates orders of magnitude less than catalysis by iron and manganese minerals. Among the predominant metal oxides, hydrogen peroxide decomposition rates were highest among the iron oxides, followed by the manganese oxide-bearing minerals. High rates of hydrogen peroxide decomposition have traditionally been correlated with the presence of manganese oxide nodules. However, these results suggest that iron oxides play a significant role in hydrogen peroxide decomposition relative to manganese oxides. Hydrogen peroxide decomposition rates were then investigated in the series of natural soils and well cuttings. The results indicate that all of the natural solids decomposed hydrogen peroxide rapidly, a phenomenon that has been documented in Fenton's ISCO field studies.

Hydrogen peroxide decomposition rates were correlated with numerous soil characteristics, such as total manganese content, total iron content, amorphous and crystalline manganese and iron content, and per cent organic matter. The results of correlation analyses indicate that hydrogen peroxide decomposition rates correlated most highly with the crystalline iron oxide content of the soils and subsurface solids ($R = 0.96$). These results suggest that, if hydrogen peroxide is to be stabilized in the subsurface for ISCO, the surface chemistry of crystalline iron oxides will need to be manipulated. Potential changes to the surfaces of the iron oxides that may stabilize

hydrogen peroxide include adding a reducing agent to change the surface characteristics or exchanging quaternary ammonium salts on the surface to decrease the surface area available for catalysis.

As part of a separate effort, significant progress has been made completing studies involving transient oxygen species generated with soluble iron. Using reactant-specific probes, the generation of hydroxyl radical, superoxide, and hydroperoxide anion has been investigated. Soluble iron-catalyzed reactions generate only hydroxyl radical at low hydrogen peroxide concentrations (<500 mg/L); hydroxyl radical and superoxide are generated at mid-level hydrogen peroxide concentrations (500 mg/L - 1%), and all three transient oxygen species are generated at high hydrogen peroxide concentrations (>1%). If iron precipitates in Fenton's reactions, either through a prolonged reaction period (>90 min) or by raising the pH to >5, hydroxyl radical generation is replaced by superoxide generation. Furthermore, if iron precipitates in soluble iron-catalyzed Fenton's reactions, hydrogen peroxide decomposition rates increase dramatically because the amorphous iron oxides formed, which are similar to the structure of ferrihydrite, rapidly catalyze hydrogen peroxide decomposition. At low concentrations of hydrogen peroxide (<0.5%), iron (II) is the most effective catalyst because the standard Fenton's initiation reaction is stoichiometrically efficient. However, when higher hydrogen peroxide concentrations are used (>0.5%), iron (II) results in rapid hydrogen peroxide decomposition; under these conditions, iron (III) is a more effective catalyst because it does not place an immediate demand on hydrogen peroxide.

Work is also being conducted on gaining a better understanding of the possible transformation pathways. For example, investigations have begun on the products found during the transformation of naphthalene by modified Fenton's reagent. A series of experiments were carried out to determine the naphthalene degradation kinetics and initial identification of products under Fenton's conditions with only the generation of hydroxyl radicals. In all the experiments the reaction was over in 15 min, and analysis showed constant naphthalene concentrations over 2 hr period. Reactions in which hydroxyl radicals have been scavenged have produced quinone, a derivative of naphthalene. Fenton's reactions generating both hydroxyl radical and superoxide have produced numerous peaks that are currently being identified. Subsequent product studies will focus on modified Fenton's reactions containing superoxide and hydroperoxide

2.2 Project CU-1289: Improved Understanding of In Situ Chemical Oxidation (Eric Hood, Geosyntec)

Key Tasks/Results

It is likely that ISCO adversely impacts intrinsic or enhanced bioremediation processes by: (1) inactivation of the indigenous microbial population through disinfection by permanganate; and (2) geochemical or metabolic inhibition of chloroethene degradation by survivor or introduced microbial populations (including bioaugmented microorganisms) resulting from the precipitation of manganese oxides (a permanganate reaction product). Data from the LC-34 site at Kennedy Space Center indicate that

permanganate treatment resulted in a minimal impact upon biomass density in the treatment group of samples relative to the control group, although a distinct shift in community composition and diversity is evident.

A variety of column experiments, and batch and continuous flow reactors are being used to establish long-term oxidant consumption estimates and kinetic parameters for permanganate and Fenton's reagent. These data will be used in conjunction with aquifer material characterization information to develop statistical correlations and predictive tools. Initial data suggests that aquifer material handling issues yield differences in permanganate consumption values, and a chemical oxygen demand test using permanganate provides a better estimate of the ultimate permanganate demand than a dichromate based chemical oxygen demand test.

A comprehensive perspective is being developed on the kinetics of groundwater contaminant oxidation by ISCO oxidants (i.e., permanganate and Fenton's reagent). An efficient protocol for measuring oxidation rates of many contaminants by permanganate has been developed using time-resolved spectrometry at an absorbance maximum of permanganate (525 nm). For Fenton's reagent, a zero-headspace reactor has been used to study the degradation kinetics of TCE and 1,1-DCE and the data fit a kinetic model that assumes one reactive species (presumably hydroxyl radical) with a steady-state concentration that is suppressed by high concentrations of reactants. Additional data gaps considered include explosives, BTEX, phenols, oxygenates, and chlorinated methanes.

2.3 Project CU-1290: Reaction and Transport Processes Controlling In Situ Chemical Oxidation of DNAPLs (Robert Siegrist, Colorado School of Mines)

Key Tasks/Results

In this project, experimental design, methods, and equipment provide for varying scales and complexities to explore peroxide and permanganate oxidation and contrasting oxidant application methods (low to high dose concentrations and low to high delivery densities) to treat different DNAPL masses and distributions under conditions representative of a range of subsurface settings, including:

Bench-scale kinetic aqueous-phase and DNAPL contaminant degradation studies were conducted yielding preliminary results in both the dissolved phase as well as the DNAPL multiphase. Preliminary dissolved phase result indicated permanganate oxidation efficiency is higher without media or NOM, with decreasing oxidant load, and with buffered pH. In addition, permanganate effectiveness is improved without media or NOM, with increasing oxidant load, and with buffered pH. Finally, Fenton's oxidation is more efficient and effective with increasing oxidant load, at lower pH, with soil present, and without NOM. Preliminary results for the DNAPL Multiphase studies indicated permanganate oxidation efficiency is higher with NOM, without goethite, and with decreasing oxidant load. Permanganate oxidation effectiveness is improved without NOM, goethite, and with increasing oxidant load. Fenton's oxidation efficiency is higher

with NOM, without goethite, and with decreasing oxidant load. Also, Fenton's oxidation effectiveness is improved with NOM, with goethite, and with decreasing oxidant load. Finally, permanganate and Fenton's reagent enhance DNAPL interphase mass transfer.

Bench-scale studies of porous media effects on oxidation reaction were conducted yielding preliminary results for slurry phase studies as well as for diffusive transport. The preliminary results for slurry phase studies indicated higher DNAPL load results in more efficient oxidation. It was also observed that the presence of goethite affects oxidation efficiency and effectiveness in both permanganate and Fenton's reagent systems. The presence of natural organic matter offered a more efficient and effective permanganate system, and a more effective Fenton's reagent system. Finally, it was noted that the interactions of variables can have key effects on system responses

Preliminary results for diffusive transport studies indicated that oxidant demand increases with increasing organic carbon content in porous media and with increasing permanganate concentration. Acrylic glass bead studies were initiated to determine NOD of groundwater, tracer/oxidant interactions, and steady-state flux of oxidant. Upscaling reaction/transport experiments were also initiated including model development and validation, and evaluation of portioning tracer validity of determining DNAPL degradation. The preliminary results from these upscaling experiments involved the development of CORT 3D (i.e., chemical oxidation reactive transport model) including differing mobile species, effective diffusion coefficients, NAPL dissolution, equilibrium or rate-limited sorption of contaminant, 2nd order oxidation of aqueous-phase contaminants, and generation of manganese oxides precipitates. In addition, intermediate 2-jD tank experiments are underway, wherein, dye tracer tests are performed to verify uniform flow.

An experimental evaluation was conducted with a focus toward coupling ISCO with pre- and post-treatment operation of surfactant/cosolvent mass recovery and bioattenuation. One phase of this evaluation involved the assessment of ISCO coupling and natural attenuation. Herein, column studies, statistical development, microbial characterizations and field soil experiments were initiated. Another phase involved an assessment of the coupling of ISCO with surfactant/cosolvent flushing. Preliminary results indicated at least 12 surfactants and 2 cosolvents reacted favorably with permanganate. Screening test results with surfactants and Fenton's reagent were less favorable due to gas and foam generation and temperature increases; however, further testing is underway. The final phase involved investigation of the viability of partitioning tracer tests (PTTs). Preliminary results indicated that tracer degradation occurs in the presence of 5, 50 and 500 mg/L permanganate. Batch tests indicate relatively low levels of permanganate result in an increase in the NAPL-tracer partitioning coefficients for PCE and TCE.

3. WEB RESOURCES

ITRC ISCO Documents

<http://www.itrcweb.org/common/content.asp?en=TA339169&sea=Yes&set=Both&sca=Yes&sct=Long>

Groundwater Remediation Technology Analysis Center ISCO Documents

<http://www.gwrtac.org/html/topics/inchemox.htm>

EPA Technology Innovative Program ISCO Documents and Links

<http://clu->

[in.org/embed.cfm?link=%2Fpublications%2Fdb%2Ftp%2Ecgi%3Ftechnology%3D2](http://clu-in.org/embed.cfm?link=%2Fpublications%2Fdb%2Ftp%2Ecgi%3Ftechnology%3D2)

<http://clu-in.org/products/chemox/>

DOE Document: “In Situ Chemical Oxidation Using Potassium Permanganate”

<http://apps.em.doe.gov/OST/pubs/itsrs/itsr167.pdf>

EPA Document: “Field Applications of In Situ Remediation Technologies: Chemical Oxidation”

<http://www.epa.gov/tio/download/remed/chemox.pdf>