

# Successful Unsaturated Zone Treatment of PCE with Sodium Permanganate

John R. Hesemann

Martha Hildebrandt

*In situ chemical oxidation (ISCO) with permanganate has been widely used for soil and groundwater treatment in the saturated zone. Due to the challenges associated with achieving effective distribution and retention in the unsaturated zone, there is a great interest in developing alternative injection technologies that increase the success of vadose-zone treatment.*

*The subject site is an active dry cleaner located in Topeka, Kansas. A relatively small area of residual contamination adjacent to the active facility building has been identified as the source of a large sitewide groundwater contamination plume with off-site receptors. The Kansas Department of Health and Environment (KDHE) currently manages site remedial efforts and chose to pilot-test ISCO with permanganate for the reduction of perchloroethene (PCE) soil concentrations within the source area. KDHE subsequently contracted Burns & McDonnell to design and implement an ISCO pilot test.*

*A treatability study was performed by Carus Corporation to determine permanganate-soil-oxidant-demand (PSOD) and the required oxidant dosing for the site. The pilot-test design included an ISCO injection approach that consisted of injecting aqueous sodium permanganate using direct-push technology with a sealed borehole.*

*During the pilot test, approximately 12,500 pounds of sodium permanganate were injected at a concentration of approximately 3 percent (by weight) using the methods described above. Confirmation soil sampling conducted after the injection event indicated PCE reductions ranging from approximately 79 to more than 99 percent. A follow-up treatment, consisting of the injection of an additional 6,200 pounds of sodium permanganate, was implemented to address residual soil impacts remaining in the soil source zone. Confirmation soil sampling conducted after the treatment indicated a PCE reduction of greater than 90 percent at the most heavily impacted sample location and additional reductions in four of the six samples collected. © 2009 Wiley Periodicals, Inc.*

## INTRODUCTION

*In situ* chemical oxidation (ISCO) with permanganate is a proven and widely used technology for the remediation of subsurface media impacted with chlorinated volatile organic compounds (CVOCs). Permanganate facilitates the rapid and complete destruction of many chlorinated and recalcitrant compounds, including perchloroethene (PCE), trichloroethene (TCE), dichloroethene (DCE), and vinyl chloride, the four contaminants of concern most commonly associated with dry-cleaner sites. At many sites, chemical oxidation can be a viable alternative to long-term, traditional remedial



technologies such as “pump-and-treat” and soil vapor extraction (SVE)/aquifer air sparge systems. Permanganate ISCO is most favorable at sites with relatively permeable subsurface conditions where it can be effectively distributed within the subsurface. However, recent advances in distribution techniques, including hydraulic or pneumatic fracturing and high-pressure direct-push injection, have made ISCO with permanganate in fine-grained soil formations more feasible.

Permanganate offers advantages over other oxidants used for CVOC destruction. It is relatively persistent in the subsurface compared to other oxidants, such as Fenton’s reagent, and does not generate the excessive heat and fugitive vapors associated with other Fenton’s-type oxidant technologies. In addition, permanganate is effective in either naturally oxidizing or reducing environments. In most cases, the subsurface environment returns to its natural state (oxidative or reductive) following the completion of oxidation reactions. This is important for sites that have been selected for a cascading remediation approach that may include the implementation of enhanced or augmented anaerobic biodegradation following ISCO.

While ISCO is frequently implemented in the saturated zone, challenges associated with achieving effective distribution and retention of permanganate or other oxidants in the *unsaturated* zone have made vadose-zone soil treatment with ISCO relatively uncommon, particularly in geological settings characterized by fine-grained (i.e., silt and clay) deposits.

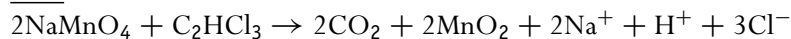
### Oxidation Chemistry

Permanganate is commercially available in two forms, potassium and sodium permanganate. The following equations give the stoichiometric reactions for the complete destruction of the four contaminants of concern using sodium permanganate:

#### PCE



#### TCE



#### DCE



#### Vinyl Chloride



Siegrist et al., 2001

As shown in each reaction equation above, permanganate oxidizes the chlorinated compound and produces carbon dioxide, manganese dioxide solids, and various ions.

### Oxidant Distribution

While ISCO is frequently implemented in the saturated zone, challenges associated with achieving effective distribution and retention of permanganate or other oxidants in the *unsaturated* zone have made vadose-zone soil treatment with ISCO relatively uncommon, particularly in geological settings characterized by fine-grained (i.e., silt and clay) deposits. The delivery of aqueous oxidant solutions, such as permanganate, in the vadose zone has been frequently conducted via lance permeation methodology. The lance permeation process consists of using a high-pressure injection wand to penetrate the ground surface to the desired depth of injection(s) and subsequently deliver the oxidant solution through the same injection wand. During the process, water or injectant exits the tip of the injection lance at pressures ranging from a few thousand to 10,000 pounds per square inch (psi). The high pressure effectively excavates a borehole that allows for the

manual insertion of the lance to depths typically ranging from 5 to 20 feet below ground surface (bgs). Since the borehole annulus is not sealed, oxidant will reach the ground surface once the annulus is full. In addition, since no pressure is maintained inside the borehole, the oxidant is not effectively forced into the soil matrix, especially a fine-grained soil matrix. To compensate for the lack of lateral distribution, lance permeation boreholes are spaced very close together, typically two to three feet.

Due to the depths of oxidant distribution required for the subject site (greater than 30 feet), an alternative to lance permeation was recommended. In addition, since the subject site is an active facility, minimizing oxidant surfacing and maintaining a relatively clean project site were important considerations. Due to advances in direct-push injection tooling, the injection of an oxidant solution under pressure within a sealed borehole was identified as a potentially feasible approach for the site. Pilot testing of the delivery approach was subsequently proposed.

This article summarizes the methods used to implement ISCO with permanganate for the treatment of CVOC-impacted soil, for the purpose of reducing source-zone concentrations to levels protective of groundwater. The ISCO implementation approach included procedures and equipment specifically designed to increase oxidant distribution, retention, and injection efficiency. The article also summarizes the results of both an injection pilot test and follow-up treatment.

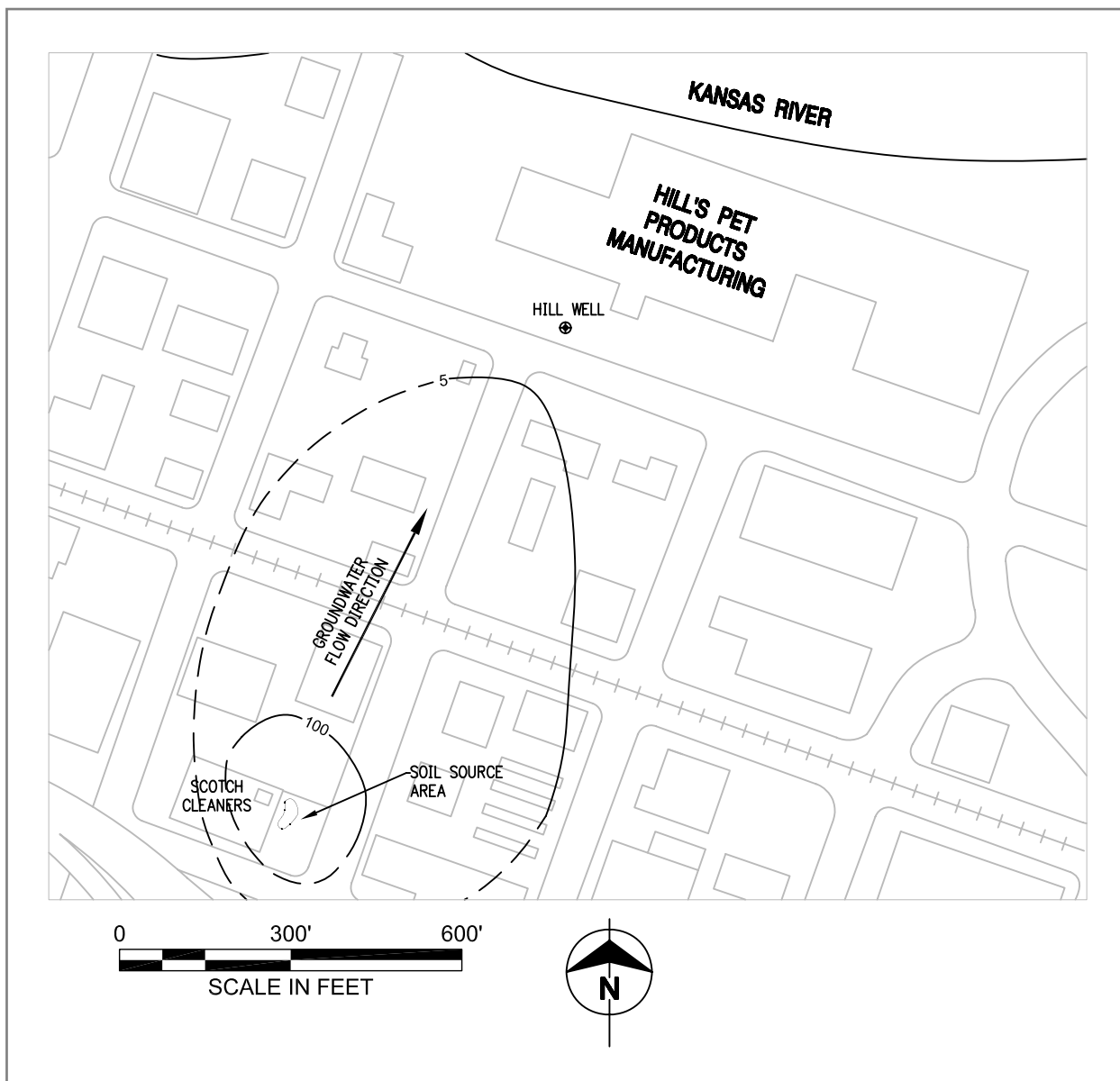
## SITE BACKGROUND

The subject site is an active dry-cleaning facility located in Topeka, Kansas. The Kansas Department of Health and Environment (KDHE) currently manages site remedial efforts and chose to pilot-test ISCO with permanganate for the reduction of PCE soil concentrations within the source area. The KDHE subsequently contracted Burns & McDonnell to perform pilot-test design and implementation, as well as pilot-test evaluation and full-scale design and implementation, if the results of the pilot indicated that the technology was feasible. The purpose of the pilot test was not only to determine the efficacy of ISCO permanganate treatment in the unsaturated zone, but also to verify the feasibility of the oxidant distribution approach.

As previously mentioned, the subject site is an active dry cleaner located in Topeka, Kansas. A pet food manufacturing facility is located approximately 1,000 feet northeast (downgradient) from the dry cleaner, and the Kansas River is located approximately a quarter mile from the site in the same direction (see Exhibit 1). Previous site investigation activities identified a relatively small area of residual soil contamination believed to be the source of the larger sitewide groundwater contamination plume. The soil source is located adjacent to the active facility building and encompasses a lateral area of approximately 1,500 square feet. Soil impacts are generally encountered from approximately 13 feet bgs to the groundwater surface, typically around 32 to 35 feet bgs. The predominant contaminant of concern, PCE, has been detected in the soil source area at concentrations ranging from 550 to 200,000 micrograms per kilogram ( $\mu\text{g}/\text{kg}$ ).

The goal of the source-area remedial action was to reduce soil-contamination concentrations to levels protective of groundwater, thereby minimizing continued groundwater plume contaminant loading. The groundwater-contamination plume associated with the dry-cleaner facility extends from the soil source area to the northeast.

This article summarizes the methods used to implement ISCO with permanganate for the treatment of CVOC-impacted soil, for the purpose of reducing source-zone concentrations to levels protective of groundwater.



**Exhibit 1.** Site layout

The majority of the plume is characterized by relatively low concentrations of PCE and trichloroethene (TCE). Concentrations in wells located within and adjacent to the source area range from approximately 400 to 2,000  $\mu\text{g}/\text{L}$  for PCE, and 400 to 4,000  $\mu\text{g}/\text{L}$  for TCE. PCE and TCE concentrations in wells located downgradient from the soil source area are generally less than 10  $\mu\text{g}/\text{L}$ . Low-level detections have been reported in a groundwater production well located on the pet food manufacturing facility property. With the downgradient manufacturing facility well constituting a groundwater plume receptor, and the Kansas River constituting a potential receptor, the KDHE sought to remediate the identified source of the plume quickly and effectively, thereby accelerating natural attenuation and overall contraction of the groundwater plume extents.

The geology at the site consists of a fining upward sequence of alluvial sediments, typical of major river valley floodplain and terrace deposits. Within the source area, silty to sandy/silty-clay soils are encountered from the ground surface to a depth of approximately 36 feet bgs. Beneath the fine-grained deposits, sandy soils extend to bedrock and coarsen with depth. Groundwater is generally encountered from approximately 32 to 35 feet bgs, and bedrock is encountered at approximately 93 feet bgs.

The KDHE considered both lance permeation and direct-push (sealed borehole) injection methods for the site. Direct-push injection was selected as the delivery method, and the KDHE determined that the site would serve as a “proving ground” for the technology. If successful, the delivery method would be considered for implementation at several other dry-cleaner sites with CVOC soil source zones requiring treatment.

## ISCO TREATABILITY TESTING

Prior to initiating the ISCO pilot test, subsurface soil samples, collected from the soil source area, were submitted to Carus Corporation for bench-scale treatability testing. Natural organic matter (NOM) and reduced metal species in the subsurface can exert a significant oxidant demand that competes with contaminants of concern for available permanganate, directly affecting permanganate persistence and transport in the subsurface, and possibly resulting in incomplete oxidation of the target compounds. In most cases, the natural oxidant demand is the most important factor in determining permanganate dosage for ISCO.

A permanganate soil oxidant demand (PSOD) study, consisting of 48-hour PSOD experiments, was performed by Carus to determine the amount of permanganate required to satisfy the site-specific PSOD. In addition to the amount of NOM, the initial dose of permanganate and the reaction time available also have a significant impact on the PSOD. Therefore, three different dosage rates (low, medium, and high) were applied during the PSOD test.

Soil samples used in the bench-scale study were collected using split-spoon sampling techniques during the installation of Monitoring Well MW-7S, located within the soil source area. One soil sample was collected from each of the two distinct soil types within the targeted treatment zone. The shallow sample (PSOD-1) was collected from 22 to 26 feet, corresponding to a silty-clay soil matrix. The deeper sample (PSOD-2) was collected from 31 to 35 feet bgs, corresponding to a silty/sandy-clay soil matrix.

PSOD bench-scale treatability study results consist of permanganate consumption values represented by mass (g) of permanganate consumed per mass (kg) of soil used in each experiment. The results of the site-specific testing indicated the following:

<u>PSOD-1 Sample</u>	<u>PSOD-2 Sample</u>
< 3.3 g/kg (low NaMnO <sub>4</sub> dose)	2.1 g/kg (low dose)
10.6 g/kg (medium dose)	6.5 g/kg (medium dose)
15.7 g/kg (high dose)	7.4 g/kg (high dose)

The average PSOD values for the two samples were 2.7 g/kg (low dose), 8.6 g/kg (medium dose), and 11.5 g/kg (high dose). The ranking system used by Carus to rate PSOD characterizes the PSOD of soils with a measured 48-hour demand of less than 15.0 g/kg as low. Due to subsurface heterogeneities that may not be reflected in the

Prior to initiating the ISCO pilot test, subsurface soil samples, collected from the soil source area, were submitted to Carus Corporation for bench-scale treatability testing.

discrete samples collected, a conservative PSOD value of 15 g/kg was used to determine oxidant dosage for the site. This PSOD value provided an adequate dosage to address potential variability within the subsurface material and contaminant concentrations.

## PILOT TESTING

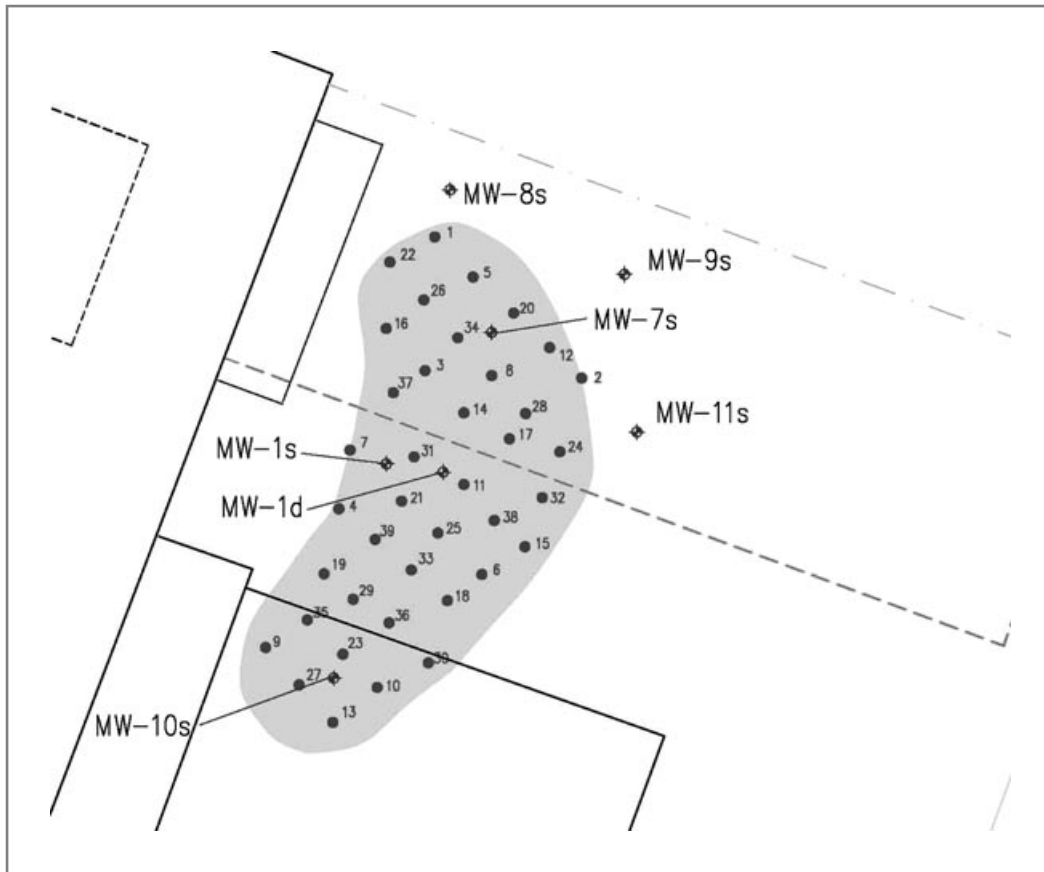
Because sodium permanganate ( $\text{NaMnO}_4$ ) is delivered from the manufacturer in a 40 percent (by weight) aqueous solution, it is easily handled and diluted with water, making it compatible with the vadose-zone "flooding" approach employed at the site.

Carus RemOx-L<sup>®</sup> (sodium permanganate) was the selected oxidant for the soil treatment application. Because sodium permanganate ( $\text{NaMnO}_4$ ) is delivered from the manufacturer in a 40 percent (by weight) aqueous solution, it is easily handled and diluted with water, making it compatible with the vadose-zone "flooding" approach employed at the site. The alternative form of permanganate, potassium permanganate ( $\text{KMnO}_4$ ), is manufactured as a 99 percent permanganate salt (solid). The solid  $\text{KMnO}_4$  must be mixed with large volumes of water to produce a diluted aqueous solution, requiring more time and labor. In addition,  $\text{KMnO}_4$  solids must be added to makeup water slowly to prevent the accumulation of solids in mixing tanks and the corresponding reduction of  $\text{KMnO}_4$  mass in the injectant solution. At the time of this field effort,  $\text{NaMnO}_4$  was significantly more expensive than  $\text{KMnO}_4$  on a mass basis (approximately 244 percent). However, for a small site, the convenience and labor cost savings associated with oxidant mixing and delivery was determined to outweigh the added material costs.

The estimated volume of vadose-zone soil treated during the pilot-test application was approximately 1,300 cubic yards. The estimated ISCO dosage for the treatment area consisted of approximately 12,600 pounds of 40 percent  $\text{NaMnO}_4$ , and the selected oxidant concentration was 3 percent by weight. The oxidant concentration was selected based on permanganate oxidation kinetics, which suggest that at concentrations above ~3 percent, the efficiency of the oxidant is diminished (i.e., increasing PSOD), and any oxidant used to increase concentration above 3 percent would effectively be wasted. Additionally, a 3 percent concentration was determined to generate a manageable oxidant solution volume, allowing the targeted permanganate mass to be delivered to the treatment area in a reasonable time frame.

The area targeted for pilot-study treatment was approximately 62 feet long by 20 feet wide, with a vertical treatment profile extending from approximately 13 to 33 feet bgs. The injection grid consisted of 39 total injection points spaced approximately two yards apart (see Exhibit 2). During the pilot-test injection field activities, approximately 500 gallons of 3 percent  $\text{NaMnO}_4$  solution were injected at each injection point. Each 3 percent  $\text{NaMnO}_4$  solution batch was prepared in a mixing tank prior to injection. Oxidant mixing and delivery were accomplished using an injection rig provided and operated by Burns & McDonnell personnel. The rig included all necessary tanks, piping, valves, pumps, and controls. The equipment setup also included a high-pressure injection pump, direct-push injection tooling, and oxidant feed and injection hoses.

The 12,650 pounds of 40 percent  $\text{NaMnO}_4$  injected during the pilot test amounted to 1,100 gallons of 40 percent  $\text{NaMnO}_4$  diluted to 19,400 gallons of 3 percent  $\text{NaMnO}_4$ . Injection was accomplished via direct-push rods, a Geoprobe<sup>®</sup> DP800 injection pump, and associated tooling. The lead direct-push rod was fitted with a standard four-port Geoprobe<sup>®</sup> injection tip to facilitate oxidant delivery at specified depths. The injection tip utilizes overburden pressure and the low permeability soil properties to develop a seal above the injection ports during probe rod advancement. During injection, high pressure



**Exhibit 2.** First injection layout

and the sealed borehole were used to force the oxidant into the fine-grained soil matrix. Tight vertical injection spacing was also used to optimize distribution.

Injection activities were conducted from September 18 through October 3, 2006. The injection program was accomplished with a field crew consisting of two injection technicians and one direct-push probe operator. The 3 percent  $\text{NaMnO}_4$  solution was injected at each location using a “top-down” method to maximize borehole seal integrity and minimize the occurrence of oxidant daylighting. Daylighting occurs when the injection pressure overcomes the strength of the borehole seal and oxidant “short-circuits” to the surface via the borehole annulus. The top-down method was implemented by initially advancing the probe rods to a depth of approximately 13 feet bgs. The prescribed volume of oxidant was injected at the initial depth, and the rods were subsequently advanced approximately two to four feet to reach the next injection interval. The process was repeated until injection was accomplished at the deepest interval (approximately 36 feet bgs). Oxidant was injected at approximately seven depth intervals at most locations, and approximately 72 gallons of oxidant were injected at each interval. Daylighting did occur at some injection locations. At these locations, oxidant was injected at approximately eight to ten depth intervals to deliver the required volume of oxidant solution.

## Sodium Permanganate Safety

The 40 percent solution also reacts violently with reducing agents, potentially resulting in an explosion that can propel the oxidant significant distances, posing an additional risk to bystanders.

The safety hazards associated with  $\text{NaMnO}_4$  have been well documented. As a 40 percent aqueous solution, the  $\text{NaMnO}_4$  provided by the manufacturer constitutes a powerful oxidant, capable of igniting dry organic materials, such as wood or paper, and causing severe burns to the skin. The 40 percent solution also reacts violently with reducing agents, potentially resulting in an explosion that can propel the oxidant significant distances, posing an additional risk to bystanders (Martin and West, 2002). Due to these hazards, Burns & McDonnell implemented several measures to minimize the chance of worker and bystander exposure to  $\text{NaMnO}_4$ , particularly 40 percent  $\text{NaMnO}_4$ . First, the area occupied by injection equipment and field personnel was cordoned off during all oxidant transfer, mixing, and injection activities. Drums of 40 percent  $\text{NaMnO}_4$  were stored in a locked, mobile storage unit staged on-site. Only one 40 percent  $\text{NaMnO}_4$  drum was removed from the storage unit at a time, and the 40 percent  $\text{NaMnO}_4$  was immediately transferred into the oxidant mixing tank and diluted to a safer concentration. All project site personnel were notified prior to opening a 40 percent  $\text{NaMnO}_4$  drum for subsequent transfer, and nonessential personnel vacated the area. During the transfer, personnel operating the transfer pump wore extensive personal protective equipment (PPE) including Tyvek<sup>®</sup> coveralls, chemical-resistant gloves, and full-face shields. While a reducing agent, sodium thiosulfate, was used to neutralize  $\text{NaMnO}_4$  during site cleanup activities, the sodium thiosulfate was always well diluted prior to making contact with  $\text{NaMnO}_4$ . In addition, the granular (nondiluted) sodium thiosulfate was stored in a building separate from the 40 percent  $\text{NaMnO}_4$  storage unit.

## PILOT-TEST RESULTS

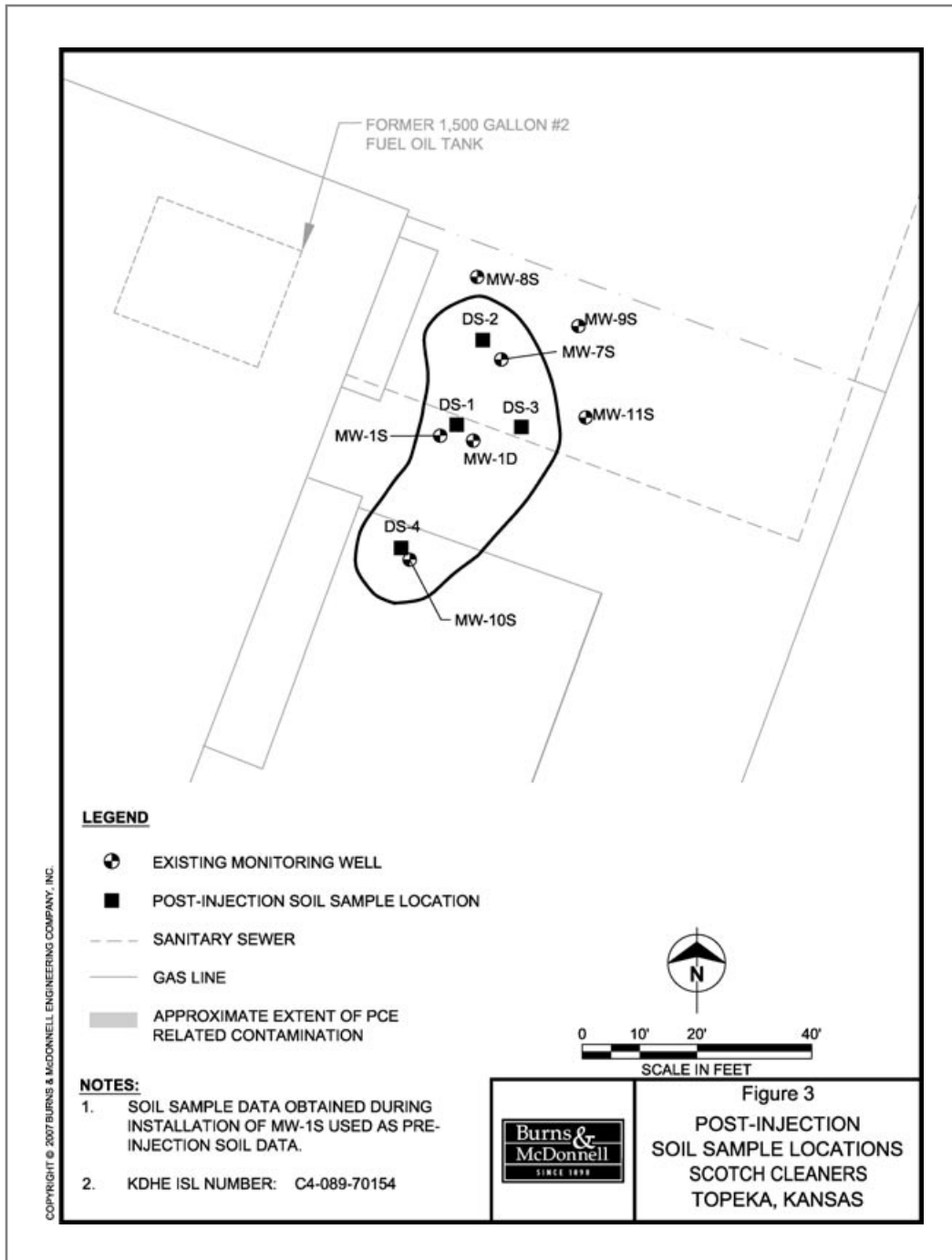
The results of the pilot test indicated that both primary objectives, effective reduction of PCE in soil and successful oxidant delivery, were achieved during the two-week injection program. The targeted volume/mass of oxidant was injected in the treatment zone, and the oxidant was evenly distributed both vertically and laterally. Oxidant daylighting occurred at 20 of the 39 injection locations. While this sometimes resulted in an injection volume that was less than the targeted volume, no significant loss of efficiency was incurred, and the appearance of oxidant solution at the surface was minimal and localized. Likely causes of daylighting include localized zones of highly impermeable soil, preferential pathways within the vadose-zone formation, and localized oversaturation caused by injection and precipitation events.

To evaluate the effectiveness of the pilot-test ISCO application, soil samples were collected within the treatment zone in February 2007 (four months following injection) and compared to baseline results from 2003 or 2005 sampling events (see Exhibit 3). Of the five confirmation soil samples collected, four indicated PCE concentration reductions of 79 percent or higher (see Exhibit 4). This included an 83 percent reduction (from 200,000 to 35,000  $\mu\text{g}/\text{kg}$ ) at the most impacted soil sample location (DS-1 [23–25 ft bgs]).

## FOLLOW-UP TREATMENT

Based on the success of the pilot test injection, a follow-up “polish” treatment was approved by the KDHE. The follow-up treatment consisted of applying ISCO to a smaller





**Exhibit 3.** Sample locations

treatment area and approximately 480 cubic yards of soil, significantly less than the 1,300 cubic yards treated during the pilot-test application.

The estimated ISCO dosage required for the treatment area was approximately 6,200 pounds of 40 percent NaMnO<sub>4</sub>, again injected at a concentration of 3 percent by weight. The area targeted for follow-up treatment was approximately 30 feet long by 20 feet wide, with the vertical treatment profile again extending from approximately 13 to 33

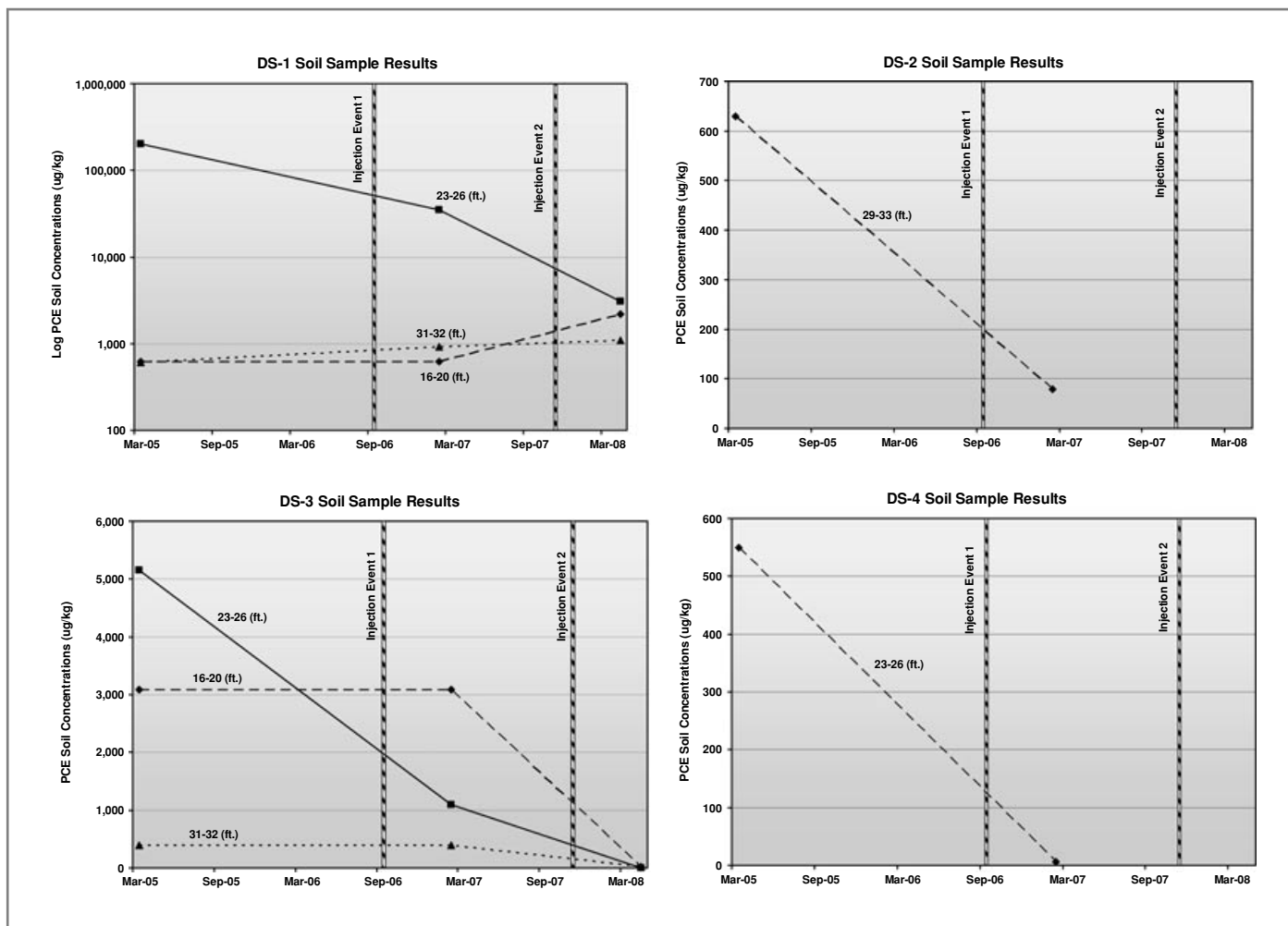
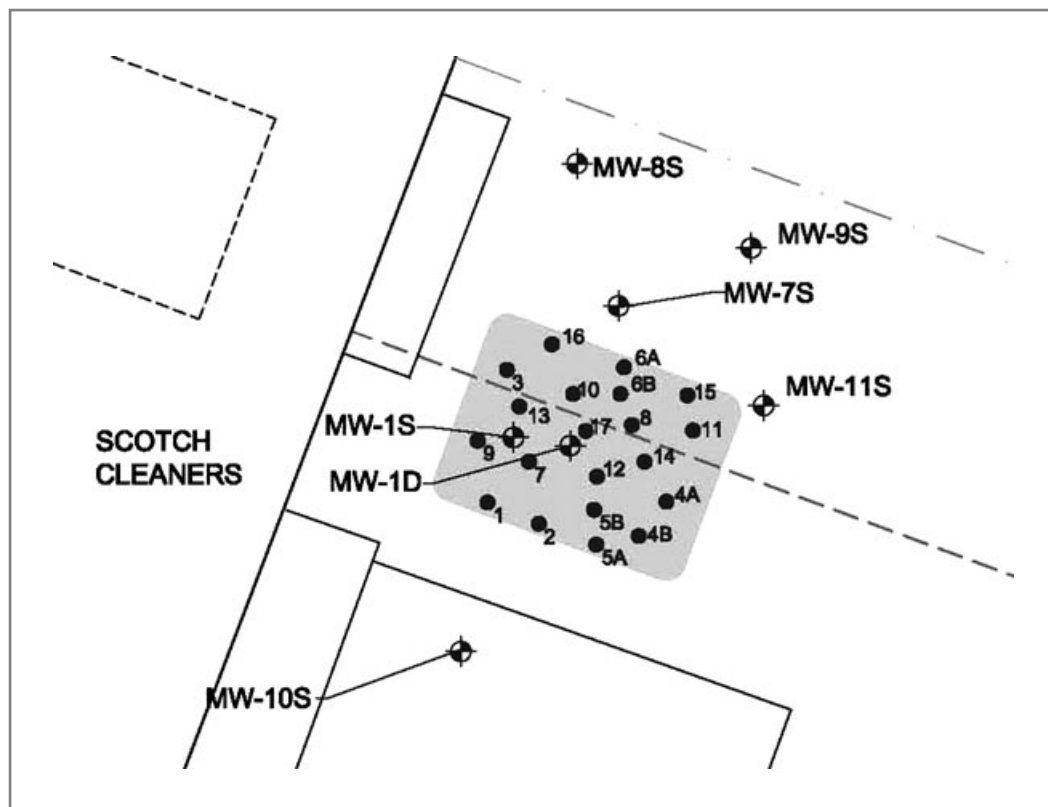


Exhibit 4. Soil data charts

feet bgs. The injection grid was slightly tighter than that used for the pilot study and consisted of 20 total injection points spaced approximately five feet apart (see Exhibit 5). The vertical injection spacing within each injection point was also tighter, with the distance between injection depths averaging about two feet and an average of 13 injection depth intervals per point. The same injection procedures and equipment used during the pilot injection were employed during the follow-up treatment field activities.

Follow-up treatment injection activities were conducted from November 6 through 10, 2007. The injection program was again accomplished with a field crew consisting of two injection technicians and one direct-push probe operator. The target oxidant volume was successfully and evenly distributed during the five-day injection period. At least a minimal amount of daylighting did occur at most injection locations, but the daylighting caused no significant loss in efficiency. The additional daylighting observed during the follow-up injection is likely attributed to preferential pathways that developed in the soil matrix during the initial injection activities.

To evaluate the effectiveness of the follow-up ISCO application, soil samples were collected within the treatment zone in April 2008 (five months following injection). A total of six confirmation samples were collected from two probe holes located within the



**Exhibit 5.** Second injection layout

follow-up treatment injection area. Samples from each probe hole were collected at three discrete depth intervals. The sample results indicated PCE reductions in four of the six samples collected (see Exhibit 4). This included an additional decrease of > 90 percent (from 35,000 to 3,100  $\mu\text{g}/\text{kg}$ ) at the previously most impacted sample location (DS-1 [23–25 ft bgs]). The two locations that did not exhibit decreases included DS-1 (15–16 ft bgs) and DS-1 (32–35 ft bgs). These locations had increases of 620 to 2,200  $\mu\text{g}/\text{kg}$  and 930 to 1,100  $\mu\text{g}/\text{kg}$ , respectively.

## CONCLUSIONS

Confirmation soil sampling results suggest that the soil source at the Scotch Cleaners site has been effectively remediated. Although residual concentrations of PCE remain in the soil, the remaining contaminant mass is likely attributed to the strong adsorption potential and relatively low solubility of PCE. That is to say, although PCE mass remains in the soil matrix, as indicated by posttreatment analytical results, it is very likely that the concentrations have been reduced to levels protective of groundwater.

The low-solubility and high-adsorption potential of PCE in particular can be demonstrated with a comparison of PCE and TCE solubilities and organic carbon partition coefficients ( $K_{oc}$ ).  $K_{oc}$  is a measure of a particular compound's tendency to partition into (or adsorb to) organic carbon (Kuo, 1999). Solubilities for PCE and TCE are 150 and 1,100 mg/L at 25°C, respectively. Measured  $K_{oc}$  values for PCE and TCE, as provided by

the U.S. Environmental Protection Agency, are 265 and 94 L/kg, respectively. In addition to the high  $K_{oc}$  value associated with PCE, the relative amount of organic carbon present in fine-grained soils is typically higher as compared to coarse-grained, sandy soils. These factors all contribute to a high potential for a nonleachable mass of PCE to remain in soils at the site following ISCO.

The tendency for detectable PCE soil concentrations to remain following the oxidation of leachable PCE contaminant mass has also been demonstrated through bench-scale column studies. Suthersan and Payne performed one such study that concluded:

These results led to the conclusion that the permanganate oxidation process would not oxidize the entire perchloroethene mass in the source area of this site, but the reduction in leaching concentration would reduce the rate of perchloroethene supply to the groundwater to levels that are protective of the aquifer for drinking water use. (Suthersan & Payne, 2005, p. 284)

Pilot testing and follow-up treatment results for this site suggest that ISCO with  $\text{NaMnO}_4$  is a viable treatment technology for vadose-zone soil remediation. The achievements of the field delivery methods suggest that challenges associated with ISCO distribution in fine-grained vadose-zone soil formations can be overcome. While residual concentrations of PCE in soil are likely to remain following ISCO, the process can be effectively implemented to reduce concentrations to levels protective of groundwater.

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**John R. Hesemann**, P.E., is a senior environmental engineer for Burns & McDonnell in St. Louis, Missouri. His expertise consists of the selection, design, implementation, and evaluation of a wide variety of environmental remediation technologies. He received his BS and MS in geological engineering from the Missouri University of Science and Technology in Rolla, Missouri.

**Martha Hildebrandt**, P.G., is a senior geologist in the environmental group of Burns & McDonnell. Her focus is in the area of investigation and remediation of federal sites. She received her BS in geology from the Missouri University of Science and Technology in Rolla, Missouri.

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