

REMEDIATION OF BTEX IMPACTED GROUNDWATER USING CHEMICAL OXIDATION – A CASE STUDY

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ABSTRACT

Underground storage tanks associated with a former gasoline station were leaking for an unknown length of time. A groundwater plume had migrated approximately 120 m down-gradient towards a residential development. The area of the off-site plume was approximately 7,300 m² and extended from depths of 5 to 12 m below ground level. Dissolved benzene concentrations were as high as 9.2 ppm (typically 5 to 8 ppm), while the remediation guideline was 0.3 ppm.

The magnitude of the off-site plume coupled with time constraints for remediation limited the remediation options available. It was determined that the most cost-effective and timely method of remediation was to use in-situ chemical oxidation.

After injecting a total of 112 m³ of hydrogen peroxide into the formation, the plume had been reduced by 90% in size, meeting the treatment objectives, and the previously elevated dissolved benzene concentrations were reduced to as low as 0.0009 ppm. Although soil treatment was not required to meet the guideline criteria, desorption of benzene was detected during the treatment period.

This paper presents a case study of the application of the in-situ remediation using chemical oxidation and the problems encountered with the implementation of this technology at depths below 10 m and in weakly cemented sand deposits.

RÉSUMÉ

Les réservoirs de stockage souterrains liés à une ancienne station d'essence fuyaient pour une quantité de temps inconnue. A la plume d'eaux souterraines a eu le vers le bas-gradient émigré d'approximativement 120 m vers un développement résidentiel. Le secteur de la plume d'au loin-emplacement était approximativement 7300 m² et étendu des profondeurs de niveau souterrain de 5 à 12 m. Les concentrations dissoutes en benzène étaient aussi hautes que 9.2 ppm (en général 5 à 8 ppm), alors que la directive de remédiation était 0.3 ppm.

L'importance de la plume d'au loin-emplacement couplée aux contraintes de temps pour la remédiation a limité les options de remédiation disponibles. On l'a déterminé que la méthode la plus rentable et la plus opportune de remédiation était d'employer l'oxydation chimique in-situ.

Après injection d'un total de 112 m³ du peroxyde d'hydrogène dans la formation, la plume avait été réduite de 90% dans la taille, répondant aux objectifs de traitement, et les concentrations dissoutes précédemment élevées en benzène ont été réduites aussi à bas que 0.0009 ppm. Bien que le traitement de sol n'ait pas été exigé pour répondre aux critères de directive, la désorption du benzène a été détectée pendant la période de traitement.

Cet article présente une étude de cas de l'application de la remédiation in-situ en utilisant l'oxydation chimique et des problèmes produits avec l'exécution de cette technologie aux profondeurs en-dessous de 10 m et dans les gisements faiblement cimentés de sable.

1. INTRODUCTION

In-situ chemical oxidation is no longer an innovative or novel technology, however its widespread application and use by environmental remediation practitioners has lagged behind other remedial technologies.

Chemical oxidation has been widely accepted by regulatory agencies, consultants and site owners who are looking to treat contaminated soil and groundwater in-situ; as a means to lower costs and minimize disruptions. However, for various reasons including cost, technological limitations and poor understanding of the practical application of the technology, other remedial alternatives are utilized.

This paper presents a case study that outlines the successful application of in-situ chemical oxidation and what factors contributed to the decision by the proponent and their consultant to select chemical oxidation as the primary remediation alternative.

1.1 Historical Use of Chemical Oxidation

Oxidation-reduction technologies, or more broadly in-situ redox manipulation (ISRM), have been documented or known to have been used to remediate over 40 sites in western Canada, including: Edmonton, Calgary, northern Alberta, British Columbia, Manitoba, Ontario, Quebec and New Brunswick. Numerous sites have also been

remediated in the United States. An accurate estimate of how widespread the technology is being employed is impossible to determine, as there is no central database of case records.

Currently, the majority of ISRM remediation programs involved the application of hydrogen peroxide (H₂O₂) as a chemical oxidant to remediate petroleum hydrocarbon contamination. Only in recent years have other chemical oxidants/reductants such as potassium permanganate (KMnO₄), sodium dithionite (NaS₂O₄) and ozone (O₃) been used to treat chlorinated solvents, heavy metals and petroleum hydrocarbons, respectively.

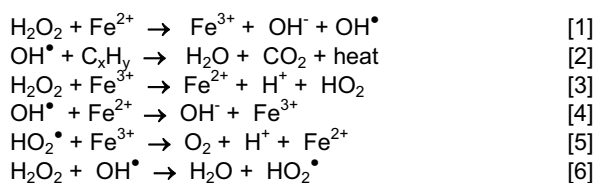
Numerous case studies can be found in academic journals, general and specialty conference proceedings, and several books. Relevant information is now available to the remediation professional whereby chemical oxidation can be evaluated for application on a specific site, without the need of advanced chemistry knowledge.

2. USE OF H₂O₂ AND FENTON'S REAGENT

For this case study site, chemical oxidation using catalysed hydrogen peroxide (Fenton's reaction) was selected as the remediation alternative. The rationale for using chemical oxidation is outlined in subsequent sections of this paper.

Chemical oxidation, as a soil or groundwater treatment technique, involves the injection of a liquid oxidant into the impacted soil or groundwater zone. The oxidant degrades organic contaminants upon contact. The reaction is non-selective, and organic molecules are oxidized in no particular sequence (Watts, 1992). This contrasts to biological degradation where the more easily degraded compounds tend to be degraded first, leaving progressively less degradable contaminants as residuals (Pardiek, et al, 1992).

While hydrogen peroxide is in itself a strong oxidant (oxidation potential = 1.8V), its oxidation potential increases significantly in the presence of mineral catalysts (2.8V), which results in both a more complete and faster decomposition of the oxidant (U.S. Peroxide 2003). Mineral catalysts include ferrous iron (Fe²⁺) and other transition metals, such as copper or manganese. Fenton's reaction is the decomposition of hydrogen peroxide by ferrous ion (Fe²⁺) which releases hydroxyl radicals (OH[•]), and it occurs through a set of primary reactions, of which some are given below (Walling 1975; Peters et al. 2001):



2.1 Known Issues When Using Fenton's Reagent

2.1.1 Safety

Hydroxyl radicals are non-specific strong oxidants, generated as an intermediate product in the aqueous phase. Hydroxyl radicals react with most organic contaminants at a diffusion-controlled rate of 10⁷ to 10¹⁰ m⁻¹ s⁻¹ (Bishop 1968). The reaction is exothermic and may be vigorous depending on the concentration of hydrogen peroxide and the organic content in the soil/groundwater matrix. This exothermic and sometimes violent reaction must be considered in detail to ensure adequate safety planning and protection for underground utilities.

Further safety considerations must include provisions for the storage of a strong oxidant at the treatment site. Typical transportation containers can include 205 L drums, totes and tankers (15,000 to 20,000 L or more). Fencing and a well-developed safety protocol are minimum requirements at the planning stage.

2.1.2 Non-Selective Reduction

Because of the non-selective nature of Fenton's reaction, the oxidant will react with any soil organic matter plus other compounds such as carbonates, thus increasing the volume of oxidant required. This becomes a significant consideration when evaluating the cost-effectiveness of chemical oxidation.

The amount of hydrogen peroxide that is required for successful hydrocarbon destruction in clay soils, for example, has been found to be 2 to 20 times more than the amount of peroxide necessary stoichiometrically (Nichols Environmental 2001) and between 5 and 50 times more in some industrial processes (U.S. Peroxide 2003). The additional hydrogen peroxide is required to overcome demand from the natural organic content of the contaminated soil (Chen et al. 1998), carbonates, and non-target organic contaminants (i.e. contaminants present but below regulatory criteria).

2.1.3 Non-Target Contaminants

Because of the non-selective nature of Fenton's reagent, consideration must be given to the concentration and distribution of all organic contaminants, not simply those that exceed a regulatory criterion. For example, at many retail gasoline fuel sites, only benzene may exceed the remediation guideline but benzene may only account for a small fraction of the organic contaminant mass. Therefore, to achieve a 99% benzene reduction a relatively similar reduction must be achieved with the other organic compounds. An oversimplification of the scope of remedial work can lead to unsatisfactory results.

It is also important to consider the total organic loading of the soil/water system when estimating oxidant requirements. For example, the remediation of groundwater impacted by a sparingly soluble organic

contaminant can theoretically only require a few litres of peroxide, but contaminants adsorbed to the soil can require thousands of litres of oxidant to remove. Without treating the soil in conjunction with the groundwater, desorption of contaminant from the soil back into the aqueous phase will occur. Although the reduction reaction is not reversible, incomplete treatment of the impacted matrix can lead to rebound and project failure.

Intermediate by-products of the oxidation reaction depend upon the contaminant, with the end product of oxidation being carbon dioxide and water. Several intermediate daughter products are formed which are typically more reactive and have a shorter half-life than the original contaminant. For example, formaldehyde is a potential daughter product of the destruction of trichloroethylene (TCE), but formaldehyde is also rapidly oxidized by H_2O_2 (Watts, 1992). This subsequent reaction is also strongly catalyzed by a conjugate reaction associated with Fenton's reagent, that being the oxidation of ferric iron. The overall result is that the original contaminant of concern is degraded or broken down to a less stable daughter product that is further degraded to harmless by-products. In practice, it is unusual or rare to find daughter products, as they are broken down before a sample can be collected and analyzed.

For many common contaminants such as BTEX, aliphatic hydrocarbons, trichloroethylene and perchloroethylene, among others, it has been shown that reaction with Fenton's reagent will not produce more toxic or non-degradable contaminants.

2.2 Practical Advantages of Chemical Oxidation

Chemical oxidation or ISRM can be used to treat both saturated and unsaturated soils, as well as groundwater. The oxidant/reductant is purchased in concentrated bulk form and is diluted with water during the injection process. The addition of water during the injection process increases the soil moisture content, which greatly enhances the effectiveness of the remediation program. It also allows the concentration of the chemical to be varied during the injection process.

The initial concentration and type of chemical oxidant applied is based on the type of contaminant and measured concentrations. The application rate is often determined through bench scale studies.

Some of the advantages of ISRM include:

- Generation of very low volumes (if at all) of remediation wastes compared to groundwater recovery or soil excavation;
- There is reduced risk of human exposure to contaminated media;
- It is less intrusive, since existing structures can be maintained (injections can be done within and below buildings);
- The technology can be applied to all or part of a given site, depending on site conditions and clean-up objectives;

- It can be used in conjunction with, or as a follow-up to other remediation techniques; and
- There are lower overall remediation costs than those associated with more ex-situ/disruptive remediation technologies.

3. SITE DESCRIPTION AND SETTING

The northern Alberta site consisted of a commercial retail complex with a former gas station on the west portion of the site, and an undeveloped parcel of land that was intended for residential development to the east. The two areas were separated by a lane. Local roads were located to the north and south and a major arterial road was to the west.

For reasons of confidentiality, the site location and owners cannot be named.

To the east, north and south, the surrounding area was primarily commercial and retail, with some high density residential. Residential areas were located to the northeast and east.

A major river was located to the east, approximately 1 km from the site and flowed south to north. The project site was located between 20 and 30 m above river elevation. The river likely acted as a discharge point for the regional groundwater but the river stage was unlikely to influence the groundwater flow regime.

Background groundwater parameters are summarized in Table 1.

Table 1: Representative Groundwater Parameters

Parameter	Units	Monitoring Well		
		MW-H	MW-10	MW-26
pH @19 °C		6.50	6.51	6.64
Conductivity @25 °C	µS/cm	1030	1060	1270
Total Organic Carbon	mg/L	17	9	11
Total Alkalinity	mg/L	430	356	507
Total Iron	mg/L	5.20	0.317	0.530
Dissolved Iron	mg/L	1.79	<0.005	<0.005
Total Manganese	mg/L	0.282	0.335	0.168
Dissolved Manganese	mg/L	0.276	0.326	0.159
Redox Potential @26°C	V	0.362	0.365	0.359

* Total Alkalinity as $CaCO_3$ to pH 4.3

Soil conditions were very uniform across the remediation area and consisted of fine-grained sand. No gravel, silt or clay inclusions or stringers were noted during drilling. The sand was found to be weakly cemented below approximately 5 m depth. The surface cover was a mixture of topsoil and former asphalt roadways.

This sand was found to extend to at least 20 m depth. Bedrock was not found within the investigation depth and

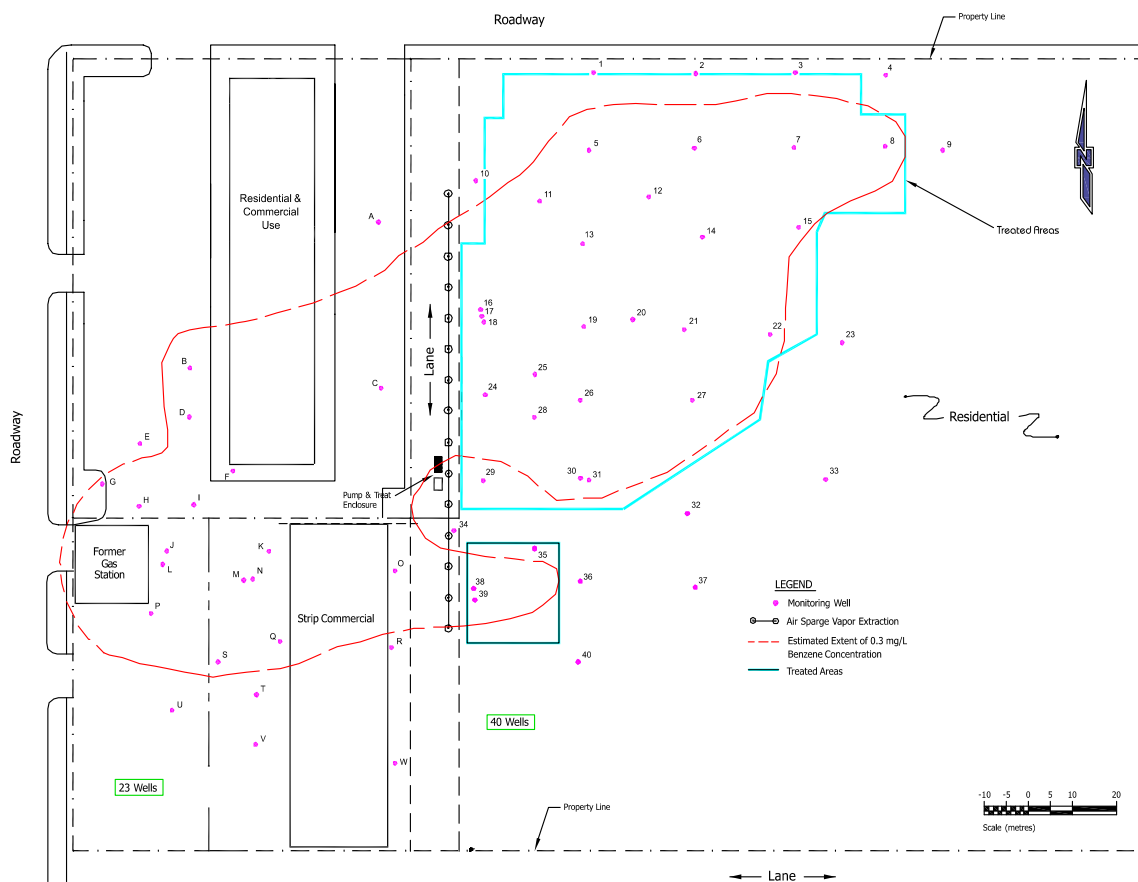


Figure 1: Site Plan Showing Dissolved Benzene Plume and Treated Areas

did not influence the remedial program. This sand was likely a post-glacial valley infill, with the groundwater flow being contained within the buried valley, with eventual discharge on the current river bank.

4. CASE STUDY BACKGROUND

Several environmental assessments had been conducted on the commercial property with minimal work being done to the east. As shown on Figure 1, 23 monitoring wells were located on the western portion of the site, and 40 were on the eastern side. However, only 4 of the 40 eastern wells were present at the start of the remediation program, with the others being installed while work was underway.

Approximately five years prior to the current remediation program, a pump & treat groundwater system was installed approximately mid-way east to west along the plume length, within the lane way. At that time the extent of contamination to the east was not known, but was assumed to be minor in extent and that the pumping system would be able to contain the plume and remediate the downgradient impacted areas.

Due to changing economic conditions the property owner of the vacant property wished to remediate the lands in order to construct various high density multi-family residential buildings. This same corporation owned the commercial property (i.e. the source site) to the west.

During the course of a due-diligence process to obtain financing for this new development, the contamination was identified. The service station owner and their consultant initiated further investigations and developed remedial alternatives.

4.1 Remediation Requirements

As with many situations dealing with third-party property owners, the potential of litigation became a significant driving force to remediate the property. This was in addition to the Provincial regulators.

Overall, the service station owner and property owner agreed that remediation of the eastern portion of the lands to the applicable residential remediation criteria would be acceptable. However, the time frame for remediation was very short. Remediation of the impacted commercial lands was the subject of further on-going discussions and was not part of this program.

At the start of remediation for the eastern lands, only groundwater required remediation.

Remediation options that were considered included: sparging with soil vapour extraction; pump & treat; excavation; and in-situ chemical oxidation.

Sparging with vapour recovery was considered; however, the time frame to complete the remediation was too long.

Pump & treat was considered, however, due to historically poor performance on this and other sites, this method was not considered viable or practical given the time constraints.

Excavation of the impacted groundwater followed by aeration of the soil/water mixture was considered as an emergency, last-ditch measure. The cost of excavation was about 10 times higher than the chemical oxidation methodology.

In-situ chemical oxidation was considered. The advantages of this method, as generally outlined in Section 2.2, and relatively moderate cost made this a comparable strategy to the next best alternative of sparging. However, chemical oxidation could be completed in less than 1/10 the time.

Based on this, a remediation program was designed. Planning, costing and scheduling were based on bench trial results and preliminary drilling information. A field trial was conducted which was turned into a full-scale remediation program within days of receiving positive laboratory results.

5. REMEDIATION PROGRAM

The overall impacted zone east of the lane was estimated to be approximately 7300 m² in size (Figure 1). The impacted groundwater zone was estimated to extend from immediately above the groundwater table at 7.2m to 9.1m.

Based on the provided data, it was calculated that up to 190,000 litres of 50% strength H₂O₂ would be required to treat the groundwater plume, assuming an average dissolved benzene concentration of 6 ppm.

5.1 Bench Trial

A bench trial was conducted to determine (1) the optimal hydrogen peroxide dosage, (2) if iron amendment was required, and (3) to evaluate if a peroxide with a reaction inhibitor was effective.

An inhibitor was considered as a means to allow greater lateral movement of the hydrogen peroxide before reacting with the contaminants. The intent would be that fewer injection points would be necessary.

Overall, the trial showed that a 94.4% BTEX reduction was achieved with a 15% strength H₂O₂ mixture (Table 2).

A very low total organic carbon concentration was measured (Table 1), which indicated that there was little naturally occurring organic matter in the aquifer which would compete with the oxidation of the identified BTEX plume.

Lower reductions were found with the 20% peroxide dose, because the reaction became vigorous and violent, expelling hydrogen peroxide from the test apparatus.

Table 2: Bench Trial on Groundwater (MW-H)

Parameter	Initial Conc.	10% H2O2	15% H2O2	20% H2O2	10% H2O2	15% Super D	15% Super D
Iron Added		No	No	No	Yes	No	Yes
Benzene	6.0	0.8	0.584	0.814	0.335	2.8	1.8
Toluene	0.24	0.12	0.023	0.027	0.017	0.029	0.029
Ethylbenzene	0.66	<0.001	0.079	0.05	0.036	0.13	0.1
Xylenes	0.88	0.336	0.238	0.30	0.39	0.52	0.43

Initial Dissolved Iron Concentration 1.79 mg/L

H2O2 indicates regular technical grade hydrogen peroxide.

Super D is technical grade H2O2 with an inhibitor to delay the reaction.

One injection only.

Lower reductions were measured with the inhibited peroxide trial. It was noted that the reaction proceeded too slowly for use in a field application. The cost of the inhibited peroxide did not warrant its use.

The bench trial further confirmed that reductions were significantly higher when iron was added to the water. The groundwater was found to have very low to non-detectable levels of dissolved iron (Table 1), which is required to catalyze the H₂O₂ reaction.

Iron additions were added to the remediation program scope prior to the injection of hydrogen peroxide.

5.2 Field Trial

A field trial was initiated around monitoring wells MW-16, MW-17 and MW-18 in August 2001, which was the most contaminated area based on the assessment work done to date.

Sampling of the three wells following treatment indicated reductions in BTEX contaminants, and in particular, the benzene concentration to levels below the established remediation criteria of 0.3 mg/L (Alberta PST Residential criteria for groundwater in coarse-grained soils, AENV 2001).

Based on these favourable results a full-scale remediation program was approved and immediately implemented.

The injection system employed on this site utilized labourers to insert injection lances into the ground with water pressure used to cut the soil. During the field trial, it was discovered that the sand was weakly cemented and that the water pressure was insufficient to break through

the cemented layers. This was not identified during auger drilling, likely because the relatively large and robust drilling and sampling equipment was able to easily penetrate the sand, which consequently destroyed evidence of cementation. This issue was overcome by pre-drilling all injection points with a solid stem auger (150 mm diameter) to the desired injection depths. Given the scale of the remediation project this additional cost was less than 2 percent of the overall budget.

5.3 Full-Scale Remediation Injections

Figure 2 depicts the injection locations, which were on an approximately 6 m spacing across the property. The spacing was adjusted to avoid asphalt roadways and surface obstructions.

Groundwater and off-gas monitoring was conducted throughout the remediation program which was undertaken from September to the end of October 2001. By October, the plume had been reduced to approximately 2700 m², down from the original 7300 m².

Based on groundwater analytical results from the end of October, there appeared to be a small localized plume around monitoring wells MW-16, MW-17 and MW-18. Soil analytical data indicated a significant variation in benzene concentrations across a large vertical zone. Based on this information, the consultant advanced boreholes to depths

up to 9.8 mBGL. As a result of this deeper drilling program, a larger soil plume was detected at depths greater than 9.1 mBGL (the original proposed injection depth). The soil plume was likely causing dissolved contaminant concentrations to rebound within this zone. As hydrogen peroxide was never injected into this deeper contaminant mass, it had not been impacted by the remediation of the groundwater above.

Subsequent drilling data indicated that there may be a highly conductive sand zone channel running up the center of the plume, which may have downward vertical gradients. The drilling confirmed that contamination within the channel area extended to depths of greater than 12 m.

Based on these revised drilling results, injections were conducted to depths of 12.5 m at the west end of the plume, within the suspected channel, near the sparge cut-off wall.

6. MONITORING AND CLOSURE SAMPLING

6.1 Field Monitoring

Throughout the process of the injections, the consultant collected groundwater samples and completed additional delineation drilling to confirm the presence or absence of BTEX contamination.

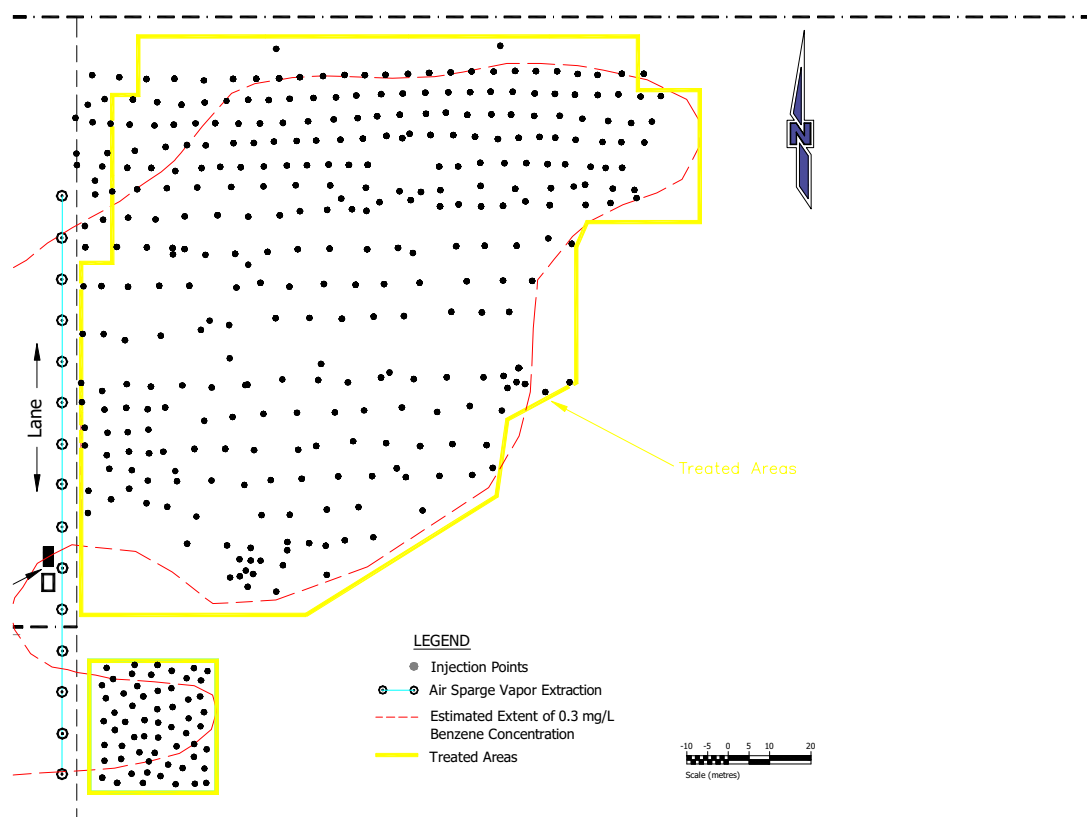


Figure 2: Injection Points

In addition, regular monitoring of carbon dioxide (CO₂), carbon monoxide (CO) and oxygen (O₂) vapour concentrations were measured in each of the monitoring wells. The effectiveness of the chemical oxidation reaction process can be monitored by measuring the CO, CO₂ and O₂ concentrations in the off-gas.

During remediation, low CO and high CO₂ and O₂ concentrations were measured throughout the remediation program. The levels of CO₂ continuously exceeded 10,000 ppm, while the oxygen concentrations were greater than 40%, which were indicative of complete oxidation of the contaminant.

At no time was there evidence of the chemical reaction short-circuiting and producing CO. High CO concentrations would indicate poor oxidation and problems with the reaction.

6.2 Groundwater Analyses

Confirmatory groundwater analyses data from representative wells is presented in Table 3 and Figure 3.

The groundwater data showed that the dissolved benzene concentration varied after each injection round. This variability demonstrates the complexity of the soil/water system.

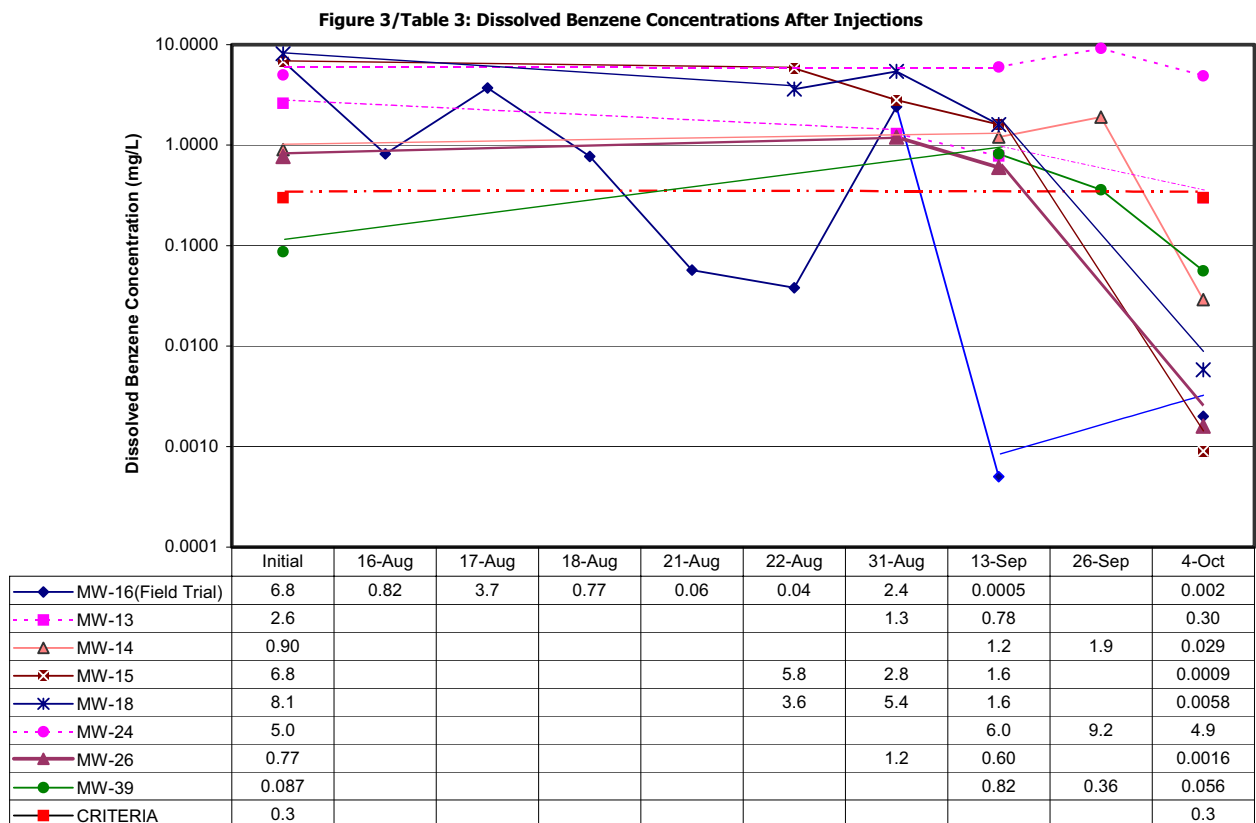
Following each round of injections, reductions in BTEX concentrations were found, as expected. However,

rebound (increases in dissolved BTEX concentrations following initial decreases) was measured in many wells. This was traced back to the contaminants adsorbed onto the soil, which dissolved back into the groundwater. The desorbed contaminant was then oxidized during a subsequent treatment pass. A typical pattern was to see contaminant concentration decrease one or more orders of magnitude, followed by a slightly smaller magnitude rebound.

This continued until the adsorbed contaminant mass was sufficiently destroyed such that the final desorbed mass would not result in groundwater exceeding the required criteria.

The volume of hydrogen peroxide and similarly the number of injection passes required to achieve remediation varied primarily based on the contaminant mass in the soil, rather than only on the dissolved contaminant concentrations. This highlights the significant planning pitfall of only considering the groundwater contaminant mass rather than the overall soil-water system. A lack of consideration to the overall contaminant distribution has led to the appearance of rebound on numerous sites and to the incorrect conclusion that an oxidation reaction is somehow reversible.

Of particular note is the continued presence of benzene above the remediation criteria in Monitoring Well MW-24 (Table 3). Due to cold weather, injections could not be completed at the end of 2001, due to the late discovery of



soil contamination at greater than 10 m depth in this area. The remaining plume area was estimated to be 500 to 800 m².

7. CONCLUSIONS

This case study highlights some of the key aspects in conducting a large-scale in-situ chemical oxidation remediation program. The results demonstrate the validity of the technology for treating slight to moderately impacted groundwater within a very short time frame and for relatively low cost compared to other alternatives.

Several difficulties in applying chemical oxidants at depths exceeding 12 m using hand-driven injection tips were overcome by pre-drilling the injection holes.

A key component of the remediation program was the continued use of field screening of oxidation off-gas to monitor the reaction process and laboratory analyses of the treated groundwater to gauge remediation progress and effectiveness. Inadequate monitoring would have resulted in ineffective use of chemical oxidant, incomplete reaction and wasted time. Frequent reviews by field and project management staff of the monitoring results allowed for rapid modifications to the treatment program, which resulted in cost and time savings.

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