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# TREATMENT OPTIONS FOR REMEDIATION OF 1,4-DIOXANE IN GROUNDWATER

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## ABSTRACT

The solvent stabilizer 1,4-dioxane has emerged in the environmental engineering arena as an unexpected and recalcitrant groundwater contaminant at many sites across the US. Decreases in the analytical detection limit in water have revealed the presence of this contaminant in sites where no 1,4-dioxane was identified during earlier, higher MDL sampling events. Toxicological studies suggest that 1,4-dioxane may be harmful, and it has been designated as a probable human carcinogen. However, the toxicity of 1,4-dioxane is in dispute and the United States Environmental Protection Agency is in the process of reviewing the toxicological information on 1,4-dioxane towards potentially revising the oral cancer slope factor and associated risk screening levels. Chemical characteristics of 1,4-Dioxane, such as high mobility, enable it to migrate much further than the solvent from which it likely originated. This has challenged remedial project managers to redesign treatment systems and monitoring networks to accommodate widespread contamination. This paper summarizes the fate and transport characteristics of 1,4-dioxane and presents current thinking in the environmental engineering community related to remedial technologies that may be applicable to groundwater treatment. At this point in time, *ex-situ* remediation has been performed at numerous sites for 1,4-dioxane, but no full scale *in-situ* remediation projects have been completed.

## INTRODUCTION

1,4-Dioxane was historically used as a stabilizer in chlorinated solvents, mainly for 1,1,1-trichloroethane (TCA) (United

States Environmental Protection Agency [U.S. EPA], 2006). The solvent TCA is regulated as a hazardous waste and was the primary focus of contaminant investigation programs, however, 1,4-dioxane is not one of the U.S. EPA's priority pollutants and does not have a maximum contaminant level (MCL) for drinking water. Consequently it has not been routinely analyzed in groundwater at solvent release sites or included in the cleanup objectives of regulatory orders (Mohr, 2001). Even sites where full Appendix VIII (40 Code of Federal Regulations Part 261) analyses were run in the 1980s or 1990s may not have identified lower levels of 1,4-dioxane because detection limits at the time were higher than they are now (e.g., 100's of ug/L). Because many 1,4-dioxane plumes are below 100 µg/L, this chemical would have been missed during characterization and remediation. Given the present uncertainty regarding the toxicology for 1,4-dioxane, it is not clear whether values as high as 100 ug/L would be harmful.

## CHEMICAL BACKGROUND

1,4-Dioxane (also known as dioxane, p-dioxane, diethylene ether, diethylene dioxide, and glycol ethylene ether (U.S. EPA, 2007a) is currently used as a solvent in chemicals (paints, lacquers, varnishes, varnish removers, wood pulping, fats, oils, waxes, and resins) and as a laboratory reagent. It may also be found at trace levels in cosmetic products such as shampoos and bath preparation, and in detergents (Agency for Toxic Substances and Disease Registry, 2007). However, it should not be confused with dioxin, an entirely different and well-known class of chemical compounds.

Stabilizers such as 1,4-dioxane were added to solvents to serve as antioxidants, acid inhibitors, and metal stabilizers, which inhibit reactions that otherwise lead to the deterioration and ultimate breakdown of the solvent, diminishing or preventing the proper solvent performance in the intended application (Mohr, 2004). Historically, 1,4-dioxane has been included with 1,1,1-TCA in mixtures at 2 to 8 percent by volume (Mohr, 2001).

The chemical structure of 1,4-dioxane consists of a cyclic organic compound with two opposed ether linkages. Its two oxygen atoms make it hydrophilic and infinitely soluble (miscible) in water. The chemical properties of 1,4-dioxane and a 2-dimensional structure diagram are shown in Table 1.

The hydrophilic nature of 1,4-dioxane makes it fully miscible in water. Because of its high solubility, 1,4-dioxane is very mobile and only weakly retarded by sorption during transport. 1,4-Dioxane's solubility in water and moderate vapor pressure may result in potential volatilization, but transfer from water to air is negligible. 1,4-Dioxane is not typically degraded by indigenous soil microorganisms under ambient conditions. Due to its infinite solubility, resistance to biodegradation under natural conditions, low Henry's Law constant, and low affinity for soil organic matter, 1,4-dioxane is extremely mobile, moving far ahead of the volatile organic compound (VOC) plumes in which it is found (Mohr, 2004).

## RISKS AND REGULATIONS

Because 1,4-dioxane is among the most mobile and persistent organic compounds released at sites, attention to the risk associated with this compound is warranted. Little data

are available related to human exposure to 1,4-dioxane, and no adequate epidemiological data are available to assess the carcinogenicity of 1,4-dioxane to humans (Mohr, 2001). International Agency for Research on Cancer (IARC) classifies 1,4-dioxane as Group 2B, possible human carcinogen based on lack of human evidence, sufficient evidence in animals, and inadequate evidence in short-term tests. The U.S. EPA classifies 1,4-dioxane as B2, a probable human carcinogen, based on the induction of nasal cavity and liver carcinomas in multiple strains of rats, liver carcinomas in mice, and gall bladder carcinomas in guinea pigs (U.S. EPA, 1995). These data show that, via the oral route, 1,4-dioxane targets the liver and nasal cavity in rats, resulting in a cancer slope factor of  $1.1 \times 10^{-2}$  milligrams/kilogram/day (Mohr, 2001). The relevancy of nasal cavity tumors to human exposure is questionable, since rats supplied with water from bottle tubes were observed to aspirate water directly into their nasal passages, a route not replicated in human ingestion. Damage to the liver was only observed at very high doses exceeding the rat's capacity to expel 1,4-dioxane. Other studies suggest that the current cancer slope factor used by the U.S. EPA significantly overestimates the potential cancer risk, and that other modeling studies may provide more accurate means for estimating potential human cancer risks (Stickney et al., 2003). Due to uncertainties regarding the toxicological studies used to derive the cancer slope factor, the severity of impact from 1,4-dioxane is currently being reassessed under the U.S. EPA's Integrated Risk Information System (IRIS) (U.S. EPA, 2006); however, the results are not anticipated to be available to the public until October 2008 (U.S. EPA, 2007b).

Based on the existing risk information and because analytical methods have improved to allow detection of 1,4-dioxane at levels similar to other VOCs, some states have now established enforceable cleanup goals. Colorado was the first state to establish an enforceable standard of 6.1 µg/L for 1,4-dioxane in groundwater after March 2005 and a 3.2-µg/L limit after March 2010 (Colorado Department of Public Health and Environment, 2005). The present standard was derived based on toxicological information presented in IRIS, however, in response to public concerns, as well as the uncer-

tainty in the toxicological research, CDPHE conservatively included the stepped down criteria in the future. It is not clear whether future changes in IRIS will be reflected in CDPHE's standards. In addition, three U.S. EPA regions and 13 other states have developed non-enforceable guidance criteria for characterizing and remediating 1,4-dioxane in soil and groundwater. Soil exposure standards range from 23 mg/kg in Florida for residential exposures to 29,000 mg/kg in Texas for industrial exposures. Groundwater standards range from 3 µg/L in California to 350 µg/L for industrial usage in Michigan (U.S. EPA, 2006).

## TECHNIQUES FOR 1,4-DIOXANE REMEDIATION IN GROUNDWATER

### Air Stripping

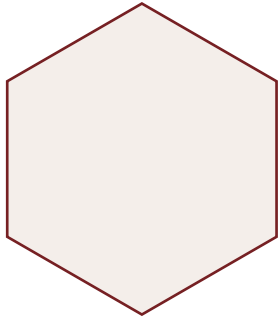
Air stripping is a typical *ex-situ* treatment for common volatile organic contaminants in groundwater, such as benzene and trichloroethene. Because 1,4-dioxane is a VOC, it is reasonable to evaluate air stripping as a potential remedial technology. However, although 1,4-dioxane has a high vapor pressure suggesting it is highly volatile, it is also highly soluble. Therefore, 1,4-dioxane is very difficult to remove from water. To evaluate 1,4-dioxane's stripping potential, air flow was optimized through a cascading water column in a 40-foot-tall packed vertical stripping tower at an ongoing groundwater remediation site. In a series of six different optimization scenarios, influent 1,4-dioxane concentrations ranged from 7.6 to 11.1 µg/L and effluent levels ranged from 7.0 to 10.0

µg/L, using air:water ratios between 183 and 291 (Earth Tech, Inc., 2004). The maximum possible removal rate approached 10 percent, which was not sufficient to meet potential cleanup standards. While this testing indicated a small decline in concentrations, the results were not repeatable or reliable enough to consider this a viable option for 1,4-dioxane remediation. A more focused and comprehensive mass transfer approach, combining air stripping with air sparging, soil vapor extraction, enhanced bioremediation, and dynamic subsurface groundwater circulation, has been demonstrated to be effective in specific circumstances (Odah et al., 2005). The in-well technology has been utilized at several sites to reduce high concentrations of 1,4-dioxane by more than 90 percent, from 28,000 µg/L to 2,400 µg/L in one case. It is not clear whether this technology would be effective for ultimately reducing levels below the groundwater standards presented above or for reductions from lower starting concentrations typical of most sites.

### Sorption

Sorption is a commonly applied *ex-situ* technology for treatment of organic contaminants in extracted groundwater. However, 1,4-dioxane has a low partitioning coefficient between soil organic matter and dissolved 1,4-dioxane in water ( $\log K_{oc}$ ) of 0.54, suggesting that it would not preferentially sorb to soil particles or other sorption media. Bench-scale treatability testing was conducted to evaluate the effectiveness of removal of 1,4-dioxane from groundwater using a variety of sorbants, including Activated

TABLE 1 Chemical Properties and Structure of 1,4-Dioxane

CAS RN	123-91-1	
Molecular weight	88.12	
Molecular formula	C <sub>4</sub> H <sub>8</sub> O <sub>2</sub>	
Water Solubility (A20°C)	Miscible	
Boiling Point (°C at 760 mm Hg)	101.32	
Melting Point (°C at 760 mm Hg)	11.8	
Vapor Pressure (mm Hg @25°C)	38.09	
Vapor Density	3.03	
Henry's Constant (atm·m <sup>3</sup> /mol)	4.80x10 <sup>6</sup>	
Log K <sub>OW</sub>	0.43	
Log K <sub>OC</sub>	0.54	
Specific Gravity (@20°C)	1.03	
Sources: CHEMFATE, 2007; Mohr, 2001		
Notes: atm = atmosphere, mm Hg = millimeters of mercury, m = meter, mol = mole, °C = degrees Celsius		

Tri-Base pelletized carbon, which contains three different types of carbon in a single pellet, surfactant modified zeolites (SMZ), SMZ with zero valent iron (SMZ/ZVI), and a proprietary macro-porous polymer manufactured by Akzo Nobel (Earth Tech, Inc., 2004). The study involved shaker tests of each medium at a range of medium:water ratios from 1:2 to 1:10,000 and at 1,4-dioxane concentrations from approximately 40 to 70 µg/L. The treatability testing demonstrated that the two zeolite formulations and the macro-porous polymer were ineffective for removal of 1,4-dioxane. Only the Activated Tri-Base carbon, manufactured by Hiatt Distributors Limited, showed effective 1,4-dioxane adsorption, but only at medium:water ratios greater than 1:20. Based on the Freundlich isotherm, a partitioning coefficient ( $K_d$ ) of 264.7 L/kg was derived, which yielded a carbon usage rate for the project of 1,479 tons per year. This quantity was far in excess of what would be cost effective or logistically achievable for this site. Research by Johns et al. (1998) looked into the sorption characteristics of granular activated carbon (GAC) sorbants derived from a variety of organic material sources. The GAC made from crushed pecan and walnut shells proved to be the most effective at removal of 1,4-dioxane from an organic chemical mixture with up to 50 percent removal rates. In addition, they identified steam activation as producing a more effective adsorbent than CO<sub>2</sub> activation of the nutshell carbon products. These studies demonstrate that sorption may be viable technology under the right site conditions (i.e., low aquifer yield, low concentrations) and with the right sorbant.

### Monitored Natural Attenuation (MNA)

Monitored natural attenuation (MNA) has been accepted as a remedial approach for many VOCs, including chlorinated solvents and petroleum contaminants, such as trichloroethene (TCE) and benzene, toluene, ethylbenzene and xylenes (BTEX), respectively. Documented attenuation mechanisms typically include biodegradation, sorption, dilution, dispersion, volatilization and chemical reactions with soil and groundwater. Natural attenuation of 1,4-dioxane has not been studied extensively, but it would be reasonable to consider some of the above mechanisms as applicable to 1,4-dioxane. Due to its high solubility and low  $K_{oc}$ , volatilization and sorption are not expected

to play a role in attenuation. Dispersion and dilution are important attenuation factors for 1,4-dioxane because it is miscible in water. Biodegradation has also been demonstrated under certain conditions and may be a factor at specific sites. Chiang et al. (2006) conducted solute fate and transport modeling using a numerical flow model to simulate observed 1,4-dioxane plume migration characteristics at an industrial site in the southeast United States. 1,4-Dioxane degradation half-lives were initially estimated based on literature reviews and a kinetic study of historical chemical data collected since the early 1990s, and then calibrated to present conditions. Several scenarios, including various degradation scenarios and a non-degradation scenario, were tested. The findings of the modeling indicated that the non-destructive scenario underestimated the decay rate observed in the field. According to Chiang et al.: "... the numerical simulation of 1,4-dioxane with a 7-year degradation half-life (corresponding to a degradation rate of 0.099 year<sup>-1</sup>) better simulated the field measurements." This modeling study was used to support the selection of MNA, which was approved by the state agency as the proposed groundwater remedy for the site.

### Phytoremediation

Phytoremediation has been demonstrated to address VOCs in groundwater, such as TCE, through mass transfer of the contaminant up through the root system and transpiration from the leaves of several different kinds of plants, as well as potential degradation of the chemicals in the root zone through enzymatic action. Phytoremediation of 1,4-dioxane was evaluated at the bench scale by Aitchison et al. (1997) using hybrid poplar cuttings. Plant uptake and destruction of contaminants in the root zone were both evaluated. An average of 54 percent of the 1,4-dioxane mass was removed from the plant reactors within 9 days. The distribution of carbon from <sup>14</sup>C labeled 1,4-dioxane indicated that the majority (77 percent) of the 1,4-dioxane removed from the reactors was volatilized, and the bulk of the remainder was present in the stem. Once released to the atmosphere, photochemically produced hydroxyl radicals can degrade 1,4-dioxane with a half-life of between 6.7 and 9.6 hours (U.S. EPA, 1995). Kelley et al. (1997) evaluated microcosm studies of *Actinomyces* CB 1190 to enhance bioremedia-

tion of 1,4-dioxane in the poplar (*Populus sp.*) rhizosphere. The bacteria were cultured in a 0.1% tetrahydrofuran (THF) solution by Dr. Rebecca Parales at the University of Iowa. Testing results indicated complete degradation of 100,000 µg/L 1,4-dioxane within 45 days. This destruction suggests that hybrid poplars, which exhibit rapid uptake of 1,4-dioxane, coupled with bioaugmentation of the root zone, could be a viable remedial technology under the right conditions. Documented field application of phytoremediation of 1,4-dioxane is limited to one study by Chiang et al. (2007). The phytoremediation system was installed in an area of approximately 8,000 sq. ft. to address a groundwater seep with the potential to impact surface water. Over 100 poplar trees were planted in 12 rows perpendicular to the groundwater flow direction. Later, an additional 100 hybrid poplar cuttings were planted between the trees to increase immediate water uptake capacity. Seep sample locations were not able to be sampled in the summer of 2006 because they were dry, which was interpreted to be a direct result of the dewatering (i.e., water uptake) capacity of the trees. Limitations of phytoremediation, regardless of the target compound, include depth to water and variable growing seasons.

### Bioremediation

Bioremediation of organic contaminants has been demonstrated to be effective at many project sites, such that it is almost considered a presumptive remedy for some contaminants, such as chlorinated organics. Laboratory research and field pilot studies have identified several different types of bacteria that are effective at either utilizing the 1,4-dioxane as a carbon and energy source or co-metabolically degrading 1,4-dioxane while consuming another carbon source. Parales et al. (1994) isolated a bacteria of the family *Pseudomonadaceae* from a sludge sample contaminated with THF that was capable of degrading 1,4-dioxane directly. These bacteria used 1,4-dioxane as the sole source of carbon, but only when it had been isolated from the THF-contaminated sludge. Direct growth of the bacteria using only 1,4-dioxane was unsuccessful. Experimental evidence indicated that more than 50 percent of the carbon in the dioxane was mineralized to CO<sub>2</sub>. Additional biodegradation evidence comes from a fixed film bioreactor that was bench tested, then field tested at the Lowry

Landfill, in Denver Colorado, on 1,4-dioxane levels of 8000 to 12,000 µg/L in groundwater (Mohr, 2004; Shangraw, 2006). The field pilot system was operated at less than 1 gpm at a controlled influent temperature of 15-25 degrees Celsius (°C) to allow direct biological destruction of the 1,4-dioxane. The site groundwater, from a former municipal landfill, was fortuitously contaminated with high levels (20,000-30,000 µg/L) of THF. The study confirmed earlier findings that the THF was necessary for the growth of the bacteria. Co-metabolic bioremediation of 1,4-dioxane has been demonstrated to be effective with several gasotrophic bacteria, including aerobic propanotrophs (Findlay et al., 2007) that destroy the 1,4-dioxane using the enzyme systems developed to utilize oxygen. In related research, the cultured bacteria (SL-D), as well as naturally occurring bacteria, can be stimulated with propane, and studies have shown that the microorganisms only destroy the dioxane after most of the propane has been consumed. The bacteria have been shown to degrade 100 percent of 1,4-dioxane, up to 10,000 µg/L, within 12 hours (Fam et al., 2005). All *in-situ* methods are subject to limitations in getting the amendment to the contaminated zones, and the groundwater chemistry alteration (e.g., anaerobic versus aerobic conditions) created to stimulate biological activity can cause naturally occurring elements to be mobilized in exceedance of drinking water standards. These changed conditions typically revert to natural conditions once the enhancing amendments are consumed.

### Chemical Oxidation

Chemical oxidation is an effective contaminant destruction method regularly applied to organic compounds such as chlorinated VOCs. Oxidation systems that have been proven effective on a variety of organic compounds in bench and field applications include ultraviolet (UV) light, ozone, hydrogen peroxide, sodium permanganate, potassium permanganate, Fenton's Reagent (H<sub>2</sub>O<sub>2</sub>+ferrous iron), UV + peroxide, ozone + peroxide, and sodium persulfate. Table 2 presents the oxidation potentials for some common oxidizers.

For 1,4-dioxane, the cyclic ether compound is more resistant to chemical breakdown and requires stronger oxidizers with an oxidation potential of greater than about 2.0 electron volts (eV). Of the

Chemical Species	Standard Oxidation Potential (volts)	Relative Strength (chlorine = 1)
Hydroxyl radical (OH)*	2.8	2.0
Sulfate radical (SO <sub>4</sub> <sup>-</sup> )	2.5	1.8
Ozone	2.1	1.5
Sodium persulfate	2.0	1.5
Hydrogen peroxide	1.8	1.3
Permanganate (Na/K)	1.7	1.2
Chlorine	1.4	1.0
Oxygen	1.2	0.9
Superoxide ion (O <sup>-</sup> )*	-2.4	-1.8

\*These radicals can be formed when ozone and H<sub>2</sub>O<sub>2</sub> decompose.  
Source: Siegrist et al. 2001

oxidants listed above, only Fenton's Reagent (H<sub>2</sub>O<sub>2</sub> + ferrous iron), UV + peroxide, ozone + peroxide, and sodium persulfate have a sufficiently high oxidation potential (eV) to reliably destroy 1,4-dioxane. The first three of these methods create the hydroxyl radical (OH), which has an oxidation potential of 2.7 eV and is identified as one of the strongest oxidizers available. Sodium persulfate has an oxidation potential of 2.1 eV, which is marginally capable, but when activated with alkaline solutions (sodium or calcium hydroxide) or steam, produces the sulfate radical (SO<sub>4</sub><sup>-</sup>), which has an oxidation potential of 2.6 eV. These higher-level oxidation methods are collectively referred to as advanced oxidation technologies and are proven technologies in *ex-situ* applications. Ozone-peroxide destruction of 1,4-dioxane in *ex-situ* applications is well documented (Bowman and Mohr, 2004; Mohr, 2004; Suh and Mohseni, 2004) as is UV peroxide (Kim et al., 2006; USACE/NAVFAC/AF-CESA, 2006; Brode et al., 2005). Schrier et al. (2006) demonstrated effective reduction in 1,4-dioxane concentrations in bench-scale studies using ozone and a combination of ozone and hydrogen peroxide. Ferrous iron (2,000 µg/L), chelated iron (2,000 µg/L iron), and alkalinity (1,000,000 µg/L as CaCO<sub>3</sub>) were added to deionized water to simulate field conditions that might be present. These additives reacted with the ozone to provide nearly as effective treatment as the ozone plus peroxide. This is a significant finding because it would eliminate the problems associated with getting the right mixture of two injected agents. While these advanced oxidation technologies can rapidly destroy 1,4-dioxane under

*ex-situ* or controlled laboratory conditions, the inherent difficulty for *in-situ* applications is getting the treatment amendment to the contaminant location. For some of the high-powered oxidizers, such as the hydroxyl radical, the very short half-life (hours) makes forced migration of the treatment materials difficult or impossible. For many oxidation approaches, the issue is further complicated by the requirement to mix two reagents, for example H<sub>2</sub>O<sub>2</sub> + O<sub>3</sub>, to achieve the high oxidation potential. In aquifer conditions, highly heterogeneous geological conditions may make accurate mixing of these amendments difficult. An additional consideration for application of oxidizers in general, is the possibility of mobilizing other contaminants, such as hexavalent chromium, which can be created by oxidizing trivalent chromium in the soil. Lastly, bromate, which has an MCL of 100 µg/L, may be formed through the oxidation of bromide, which is common in natural groundwater. In carefully controlled *ex-situ* applications, bromate production can be minimized or eliminated by optimizing the proportions of various oxidizers, but *in-situ* applications are much harder to regulate and bromate production may be an issue. *In-situ* application of chemical oxidation for 1,4-dioxane treatment has not been documented at more than the field pilot scale.

### SUMMARY AND CONCLUSIONS

The solvent stabilizer 1,4 dioxane has emerged recently as a groundwater contaminant of concern at a number of sites across the U.S., and state and federal regulatory agencies are focusing considerable attention on defining the magnitude and extent, and possible impact, of 1,4-dioxane sources and

plumes, as well as identifying applicable remedial technologies. The chemical characteristics that make 1,4-dioxane particularly troublesome, from a cleanup standpoint, are its miscibility in water, low sorption characteristics, and minimal biodegradability, making it highly mobile and environmentally persistent in groundwater.

As of the publication of this article, the only successfully demonstrated full-scale technologies for 1,4-dioxane remediation are groundwater extraction and ex situ treatment using controlled bioreactors or advanced oxidation techniques (e.g., UV+ peroxide, ozone + peroxide, etc.). Adsorption methods using specialty carbon materials indicate some level of removal that might be sufficient, but the selection of the carbon type appears to play a large role. Air stripping has been shown to be ineffective in the traditional sense, but may have applications using in-well stripping, sparging and aerobic biostimulation techniques.

Many in situ approaches have been demonstrated in bench-scale and field-scale treatability tests and show promise for full-scale application. Among these, the most promising for typical 1,4-dioxane sites with concentrations in the hundreds of ug/L, appear to be *in-situ* chemical oxidation and bioremediation. Typical limitations of any in situ method, such as delivering the oxidant to the contaminant, are exacerbated by the reactivity and short half life of the strong oxidizers required. Bioremediation methods appear to require non-native microorganisms, as well as nutrients, to achieve optimal conditions for the destruction of the 1,4-dioxane. Research programs in industry and academia are actively seeking additional chemical and biological solutions to this vexing problem.

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