Practical Perspectives of 1,4-Dioxane Investigation and Remediation

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Practical Perspectives of 1,4-Dioxane Investigation and Remediation

Sheau-Yun (Dora) Chiang

Richard (Hunter) Anderson

Michael Wilken

Claudia Walecka-Hutchison

1,4-Dioxane (dioxane) is a contaminant of emerging concern that is classified by the U.S. Environmental Protection Agency as a likely human carcinogen. Dioxane has been used as a minor or major ingredient in many applications, and is also generated as an unwanted by-product of industrial processes associated with the manufacturing of polyethylene, nonionic surfactants, and many consumer products (cosmetics, laundry detergents, shampoos, etc.). Dioxane is also a known stabilizer of chlorinated solvents, particularly 1,1,1-trichloroethane, and has been commonly found comingled with chlorinated solvent plumes. Dioxane plumes at chlorinated solvent sites can complicate site closure strategies, which to date have not typically focused on dioxane. Aggressive treatment technologies have greatly advanced and are clearly capable of achieving lower parts per billion cleanup criteria using ex situ *advanced oxidation processes and sorption media.* In situ *chemical oxidation has also been demonstrated to effectively remediate dioxane and chlorinated solvents. Other* in situ *remedies, such as enhanced bioremediation, phytoremediation, and monitored natural attenuation, have been studied; however, their ability to achieve cleanup levels is still somewhat questionable and is limited by co-occurring contaminants. This article summarizes and provides practical perspectives on dioxane analysis, plume stability relative to other contaminants, and the development of investigation tools and treatment technologies.*

OCCURRENCE OF DIOXANE

1,4-Dioxane (dioxane) is a contaminant of emerging concern (CEC) that is highly water soluble and not readily biodegraded (Mohr et al., 2010; USEPA, 2014, White et al., 1996; Zenker et al., 2003). It is classified by the U.S. Environmental Protection Agency (USEPA) as a likely human carcinogen and has been found in groundwater across the United States (USEPA, 2014). USEPA Integrated Risk Information System (IRIS) assessment for dioxane was updated on August 11, 2010, and again in September 2013 (USEPA, 2013a). It is also mentioned in a Toxicological Review document (USEPA, 2013). The 2013 IRIS assessment provides an updated oral cancer slope factor of 0.10 $(mg/kg/day)^{-1}$ based on liver tumors in female mice in a more recent study (Kano et al., 2009). It is notable that while USEPA regulates dioxane as a nonthreshold carcinogen (USEPA, 2013a, 2013), this is inconsistent with the approach taken by the World Health Organization (WHO, 2005), the European Union (EU, 2002), and by Canada (Environment Canada/Health Canada, 2010), which have determined that there is a threshold for adverse effects of dioxane, including cancer. Application of a threshold

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approach to cancer results in health-based guidelines that are an order of magnitude less stringent than the approach applied by USEPA.

Dioxane does not currently have a federal drinking water standard or maximum contaminant level (MCL) in the United States; however, USEPA Safe Drinking Water Act has dioxane listed on its 2009 final Contaminant Candidate List 3 (CCL 3), meaning it is an emerging contaminant and a candidate for the development of an MCL for the protection of drinking water quality. USEPA third Unregulated Contaminant Monitoring Rule (UCMR3) also includes dioxane (https://www.epa.gov/dwucmr/third-unregulated-contaminant-monitoring-rule). For UCMR3, all Public Water Systems (PWSs) serving more than 10,000 people and 800 representative PWSs serving 10,000 or fewer people have been monitored for 21 emerging contaminants, including dioxane, from January 2013 through December 2015. USEPA UCMR3 occurrence data indicate that dioxane was detected in approximately 22 percent of PWSs, and 7 percent of detections from PWSs exceed the reference concentration of 0.35 μg/L. Dioxane has not been detected above the 10^{-4} risk level of 35 μg/L in any of the PWSs tested (https://www.epa.gov/dwucmr/data-summary-third-unregulated-contaminant-monitoring-rule). Among the 22,610 samples collected for UCMR3, 11.5 percent of the samples (or 2,613 samples) had dioxane detections and 3 percent (or 708 samples) exceeded 0.35 μg/L. Of the 708 sampling data points, 76.3 percent of the exceedances were collected from PWS facilities using groundwater as a drinking water source, and 20.8 percent of the exceedances were collected from PWS facilities using surface water as a drinking water source (Exhibit 1).

While USEPA has not yet made the determination on regulating dioxane, it is important to note that some states have their own regulatory processes to protect drinking water resources, and the state values may change or be developed in advance of USEPA's MCL decision. Moreover, the new EPA toxicity values for risk assessment have led to much more stringent state cleanup levels. From New Jersey to Michigan, dioxane criteria can range widely (Suthersan et al., 2016). For instance, the New Jersey Department of

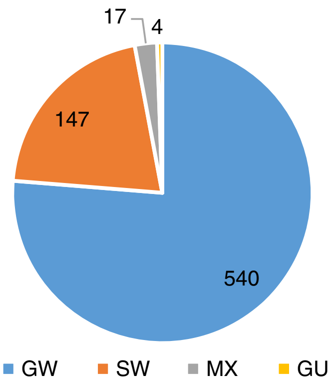

Exhibit 1. UCRM3 number of samples showing dioxane concentrations greater than 0.35 μg/L from different facility water sources. GW = groundwater (540 samples), SW = surface water (147 samples), GU = groundwater under the direct influence of surface water (four samples), MX = any combination of SW, GW, and GU (17 samples). Data source (USEPA, 2016)

Environmental Protection developed an interim specific groundwater quality criterion of 0.4 μg/L and practical quantitation limits (PQLs) of 0.1 μg/L for dioxane in October 2015. The Michigan Department of Environmental Quality is proposing a new dioxane drinking water standard change from 85 to 7.2 μg/L as it pertains to the Pall-Gelman dioxane contaminant plume in Ann Arbor, Michigan. In Europe, the German Federal Environmental Agency suggested a precautionary guideline limit for weak or nongenotoxic compounds such as dioxane at 0.1 μg/L in its Drinking Water Regulation (2001). On July 10, 2008, the Danube, Meuse, and Rhine (DMR) memorandum 2008 was established, which contains threshold values for sustainable protection of water and target values for water quality. This document also contains target values of 0.1 μg/L for component groups that have no legal target values such as dioxane, pharmaceuticals, endocrine disrupting chemicals, biocides.

Dioxane was primarily used as a stabilizer for chlorinated solvents. While its usage in the context of solvent stabilization has declined (Mohr et al., 2001), co-occurrence of dioxane and chlorinated solvents due to historic handling, storage, and disposal practices of chlorinated solvents have resulted in widespread groundwater contamination. While investigation of dioxane plumes increased since early 2000s, this effort often occurs decades after the chlorinated volatile organic compound (CVOC) plumes were investigated. The management of dioxane plumes creates a new uncertainty on top of existing challenges of managing CVOC plumes for achieving site closure. With the fact that some states do not have regulatory criteria and the regulatory criteria are often evolving, the responsible parties may choose different paths on prioritizing CVOC sites for further investigation and remediation of dioxane. The correlation between dioxane occurrence and CVOC plumes was first documented by Anderson et al. (2012) based on the U.S. Air Force's site data sets. Adamson et al. (2015) further quantify dioxane occurrence at chlorinated solvent sites using different regional meta-data sources and clearly show that 1,1,1-trichloroethane (TCA) and/or trichloroethene (TCE) account for almost all dioxane occurrence at industrial sites contaminated with chlorinated solvents, but also, respectively, underscore the lack of dioxane occurrence data at a significant number of applicable sites, particularly those with previous detections of TCA.

Adamson et al. (2015) further quantify dioxane occurrence at chlorinated solvent sites using different regional meta-data sources and clearly show that 1,1,1-trichloroethane (TCA) and/or trichloroethene (TCE) account for almost all dioxane occurrence at industrial sites contaminated with chlorinated solvents, but also, respectively, underscore the lack of dioxane occurrence data at a significant number of applicable sites, particularly those with previous detections of TCA.

In addition to the use as a solvent stabilizer, dioxane has been documented to be used in several commercial and industrial processes and also occurs as a by-product of manufacturing during the production of polyethylene teraphthalate (PET) plastic and various consumer products containing polyethoxylated surfactants (Mohr et al., 2010; Nyer, 2009; USEPA, 2013b; Zenker et al., 2003). Therefore, in some cases, dioxane is detected without the co-occurrence of chlorinated solvents. Dioxane has been monitored and treated at such industrial manufacturing sites long before dioxane was recognized to co-occur with CVOCs. Such industrial sites provide variable data for understanding of dioxane behaviors in the subsurface (Chiang et al., 2008). Based on a private survey of 76 dioxane impacted sites within the United States (20 percent government, 20 percent Department of Defense (DoD), 31 percent industry, 26 percent chemical and pharmaceutical, and 3 percent other sites), 77 percent of these dioxane sites have been impacted by chlorinated solvents, and 11 of these 76 sites have been impacted by dioxane only with no other co-contaminants (data not presented).

In Europe, dioxane is currently only produced in Germany. The production volume in 1997 was estimated to be 2,000 to 2,500 metric tons with an export outside the European Community of 575 metric tons (Romero et al., 1998). There is no information about import volumes of dioxane into the European Union (EU). The presence of dioxane was confirmed in surface waters in Germany, the Netherlands, and in the United Kingdom (EU, 2002). For instance, dioxane concentrations reached 2,200 nanograms per liter (ng/L) in the German Oder River, and 860 ng/L in the Main and Rhine River. In all rivers, concentrations of dioxane increased with distance away from springs. Dioxane was also detected in municipal landfill leachate in Sweden as well as in the industrial wastewater from polyester resin production (Paxéus, 2000; Romero et al., 1998). 1,4-Dioxane was also documented to be detected in several Dutch drinking water production locations (Ministry of Environment and Infrastructure, Netherlands, 2015). Literature also reports the detections of dioxane in the influent (262 to 834 ng/L) and in the effluent (267 and 62.260 ng/L) of four German domestic wastewater treatment plants (Stepien et al., 2014). The increased dioxane concentrations in the effluents were due to dioxane impurities in the methanol used in the postanoxic denitrification process in one of the plants. No removal of dioxane during water treatment was observed.

Dioxane's moderate vapor pressure suggests that its volatilization from dry soil is possible (Mohr et al., 2010). However, volatilization from water as well as sorption to soil organic and mineral surfaces are not expected to be significant (ADSTR, 2012, Mohr et al., 2010; Nyer, 2009; Zenker et al., 2003; see Exhibit 2). Although dioxane is relatively unaffected by sorption mechanisms, dioxane can be physically trapped into unsaturated, low permeable, and moist soils in the source area or in the fine-grained media in the aquifer. The diffusion of dioxane mass into and out of fine-grained media relative to co-released chlorinated solvents may consequently result in long-term storage and release near source areas. Back diffusion of dioxane mass can be the dominant long-term "secondary source" at many contaminated sites that must be managed (Adamson et al., 2016).

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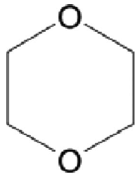
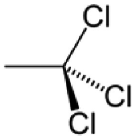
1,4-DIOXANE DATA VARIATIONS

Understanding of occurrence and extent of dioxane contamination in different environmental media has advanced with the evolution of analytical method development. A modification to USEPA Method 8260 has allowed for lower detection limits. Determination of dioxane in water at low detection levels is most often accomplished using modified USEPA Method 8270 with liquid–liquid extraction. However, according to this method, a correction for the generally lower extraction efficiency is not permitted resulting in substantially biased low data.

Recently, a comparability study based on blended, nonspiked environmental samples was conducted to evaluate the analytical results of dioxane using different analytical methods on a wide range of dioxane concentrations (Exhibit 3). The results have shown significantly lower concentrations (up to a factor of 3) when method 8270 was used compared to method 8260. The data variations have been shown for samples with a wide concentration range, different extraction methods, and even different analytical laboratories (Exhibit 3). To avoid these substantially biased data, the application of isotope dilution (using labeled dioxane as surrogate) is recommended to correct for the generally lower extraction efficiency of that method and to obtain reliable data in the low concentration range.

Exhibit 2. Physical and chemical properties of dioxane and TCA

Property	1,4-Dioxane	1,1,1-Trichloroethane	Structure
Chemical Formula	$C_4H_8O_2$	$C_2H_3Cl_3$	
CAS No.	123-91-1	71-55-6	1,4-Dioxane 
Molecular Weight	88.10 g/mol[a]	133.42 g/mol[a]	
Solubility	Miscible[a]	1334 mg/L at 25 °C[a]	
Effective Solubility	1,991,600 mg/L[a]	740 mg/L[a]	
Boiling Point	101.1 °C at 760 mm Hg[b]	74.1 °C at 760 mm Hg[c]	
Density	1.0329 g/mL[a]	1.3376 g/mL[a]	
Vapor Pressure	38.09 mmHg at 25 °C[a]	100 mmHg at 25 °C[a]	
Henry's Law Constant (K_H)	4.80 X 10^{-6} atm m^3/mol[a]	8.0 x 10^{-3} atm m^3/mol[a]	1,1,1-trichloroethane
Log K_{ow}	−0.27[b]	2.49[c]	
Log K_{oc}	1.23[a]	2.18[a]	
Estimated K_d (K_{oc} X f_{oc})	0.10[a]	15.2[a]	

[a]Mohr et al. (2010), [b]ATSDR (2012), [c]USEPA (2006).

In 2008 EPA released Method 522 for the Drinking Water Program. Method 522 determines dioxane in drinking water by solid phase extraction (SPE) and gas chromatography/mass spectrometry (GC/MS) with selected ion monitoring (SIM). Method 522 was adapted as the analytical method for dioxane analyses under UCMR3. It is worth noting that this method does not require correction for the recovery rate as well, although the rates are generally much higher than with Method 8270.

With the advancement of analytical methods and more stringent regulatory requirements, remediation managers and environmental practitioners may choose different analytical methods to achieve reporting levels lower than the regulatory requirements (lower parts per billion). The switch of analytical methods and adopting different methods for lower reporting limits and isotope dilution may lead to different but unbiased results.

1,4-DIOXANE PLUME BEHAVIORS AND NATURAL ATTENUATION POTENTIAL

Investigation of the extent of dioxane contamination has increased significantly at CVOC impacted sites in the past two decades. However, at many project sites the extent of

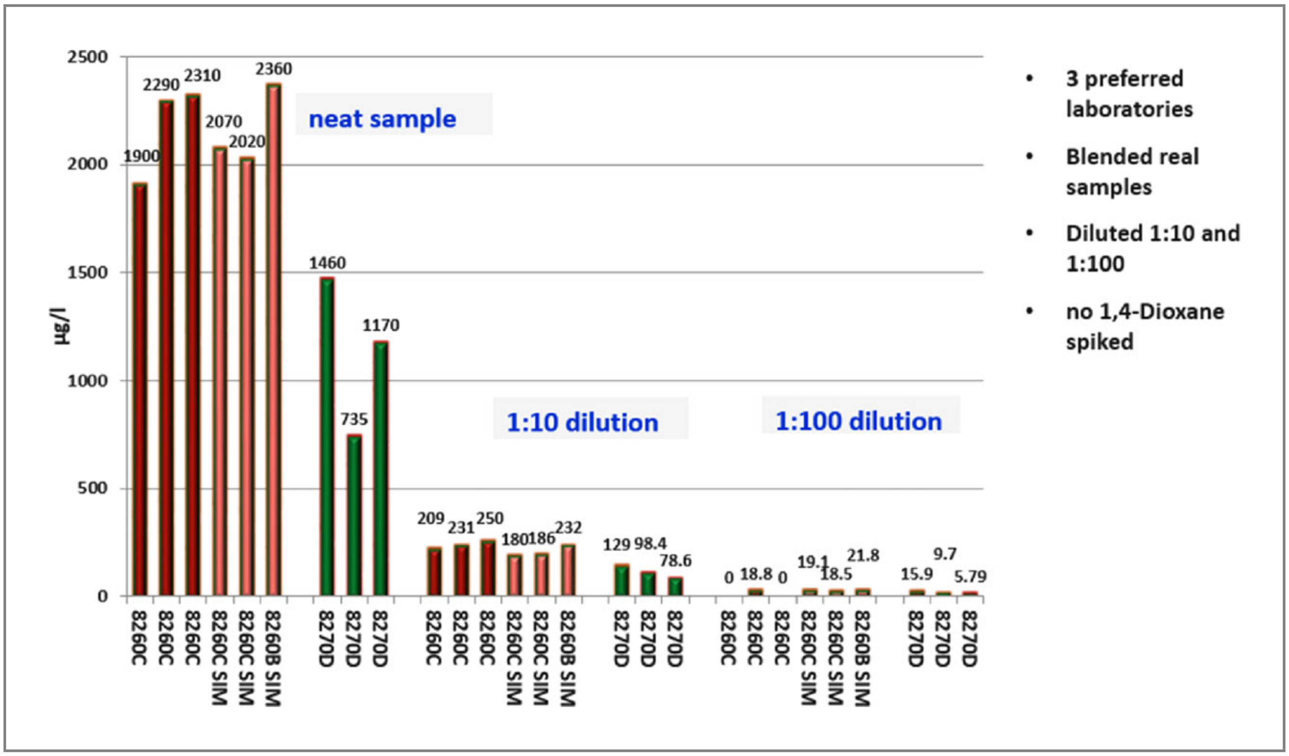


Exhibit 3. Direct comparison of dioxane analytical methods over a wide concentration range

dioxane plumes is still being investigated. The understanding of dioxane plume stability/behaviors, despite years of groundwater monitoring data, remains limited. Chiang et al. (2008) reported the potential for shrinkage of a 400-acre dioxane plume at an industrial site with no remedial measures based on more than 10 years of groundwater monitoring data. The mechanisms governing the plume shrinkage were not studied; however, natural attenuation of dioxane was likely possible. In a recent multisite dioxane survey, dioxane plume length was compared with comingled CVOC plume length and it was determined that 21 percent of dioxane plumes were larger than the chlorinated solvent plumes, 17 percent had similar plume lengths, and 62 percent showed dioxane plumes shorter than the chlorinated solvent plumes (Adamson et al., 2014, 2015). This survey further evaluates the attenuation potential of dioxane when CVOCs are co-present. It indicates that statistically significant positive attenuation rates were confirmed at 22 studied sites. At sites where dioxane and CVOCs were co-present, the median value of all statistically significant dioxane source attenuation rates was lower than TCA but similar to 1,1-dichloroethene (1,1-DCE) and TCE. This study also identifies a positive correlation between dioxane attenuation and increasing concentrations of dissolved oxygen, while the same analysis found a negative correlation with metals and CVOC concentrations. This study suggests that natural attenuation may be used to manage some, but not necessarily all, dioxane-impacted sites.

Dioxane is known to biodegrade in microcosms when a primary substrate is present to support co-metabolic biodegradation, or when dioxane is utilized by specific dioxane degrading bacteria (such as *Pseudonocardia dioxanivorans* CB1190) as the sole energy and carbon sources (Grostern et al., 2012; Li et al., 2010; Mahendra & Alvarez-Cohen, 2005;

Mahendra & Alvarez-Cohen, 2006; Parales et al., 1994; Sales et al., 2011; Shen et al., 2008; Steffan, et al., 2007). Determining whether dioxane biodegradation governs the dioxane plume shrinkage requires years of chemical data and additional lines of evidence to verify the presence, abundance, and activity of dioxane degraders. In recent years, environmental molecular diagnostics (EMDs) and compound-specific isotopic tools have been developed for validating dioxane degradation. Exhibit 4 summarizes the tools used to characterize dioxane biodegradation potential. These tools are beginning to be used by practitioners to verify natural biodegradation and performance of *in situ* metabolic and cometabolic biostimulation and bioaugmentation of dioxane plumes. It should be noted that the usefulness of some of these tools has only been confirmed in microcosms, and, therefore, their usefulness under field conditions requires further case studies for verification. Additionally, positive responses using these tools only provide secondary evidence of dioxane attenuation potential, the data can be meaningful only when plume shrinkage and declining trends are observed.

PRACTICAL REVIEW OF CURRENT TREATMENT TECHNOLOGIES FOR DIOXANE

As mentioned previously, site groundwater can be impacted by dioxane due to industrial manufacturing activities; however, data show 50 to 70 percent of dioxane sites are comingled with CVOCs. Dioxane does not respond well to several groundwater treatment technologies that are effective in treating CVOCs such as air stripping and carbon adsorption, and dioxane is therefore found escaping from CVOC treatment systems and discharged back into the environment. Exhibits 5 and 6 summarize the *in situ* and *ex situ* treatment technologies effective for dioxane treatment, respectively. The technologies that are not effective but are common for VOC treatment are also included. These exhibits can be used by practitioners as guidance when selecting dioxane technologies or when CVOC treatment technologies need to be transitioned to single or multiple technologies that can degrade both CVOCs and dioxane.

Exhibits 5 and 6 also identify the applicability and implementability of each technology for treatment of the source area, hot spots, or large dilute plume. For *in situ* technologies, *in situ* chemical oxidation (ISCO) is the most demonstrated and proven technology for treatment of both CVOCs and dioxane in the source area. Low-cost biostimulation technologies or monitored natural attenuation (MNA) may be considered favorable for a large and dilute dioxane plume, especially when the dioxane source is under remediation and there is no identified sensitive receptor to be impacted. Pilot-scale cometabolic biodegradation of dioxane with propane injection and bioaugmentation in the source area has also been demonstrated in the field, but no full-scale treatment has been reported (Lippincott et al., 2015). *In situ* metabolic biodegradation treatment of dioxane is limited to an *in situ* microcosm study in which a biotrap device baited with dioxane degrader CB1190 was deployed at a monitoring well to characterize the CB1190 abundance on the biotrap in a natural nonamended environment (manuscript in preparation). It is unlikely that dioxane would be biodegraded under anaerobic conditions. Studies have been conducted to evaluate whether methane generated from the ERD process can migrate downgradient to the aerobic aquifer and be utilized by methane

Exhibit 4. List of advanced tools for verification of dioxane biodegradation

EMD	Description[a]	Application on 1,4-Dioxane	Reference
qPCR	A laboratory analytical technique for quantification of a target gene based on DNA. Readily and commercially available for some key organisms and biodegradation associated genes Data are used to confirm the presence and determine the abundance of target microbes and genes, estimate total microbial numbers, and allow monitoring of population growth and the distribution of microbes involved in bioremediation	Use of tetrahydrofuran monooxygenase and propane monooxygenase genes of strains ENV478 and ENV425 for cometabolic biodegradation, with THF or propane as primary substrate Use of phenol hydroxylase (PHE), ring-hydroxylating toluene monooxygenase (RMO), toluene dioxygenase (TOD), methanotrophs (MOB) and soluble methane monooxygenase (sMMO) for evaluating the MNA potential of a large dilute dioxane plume Soluble di-iron monooxygenase (SDIMO) genes development and demonstration in arctic groundwater impacted by dioxane Development of 1,4-dioxane biomarkers (DXMO and ALDH) applicable for both microcosm studies and field samples. DXMO and ALDH are commercially available	Steffan, 2007 Chiang et al., 2012 Li et al., 2013 Gedalanga et al., 2014
RT-qPCR	A laboratory analytical technique for quantification of a target gene based on RNA. The data provide indirect evidence of microbial activity by detecting expression of biodegradation associated genes. Field procedures can cause RNA decay, data are generally not quantitative	1,4-Dioxane biomarkers (DXMO and ALDH) applicable for both microcosm studies and field samples	Gedalanga et al., 2014
SIP	A synthesized form of the contaminant containing a stable isotope (such as ^{13}C label) is added. If biodegradation is occurring the isotope will be taken up by the organism and detected in biomolecules (e.g., phospholipids, DNA) or respired CO_2. By tracking ^{13}C label contaminant, it is possible to obtain direct evidence of biological degradation of contaminants, and identify the degrading microorganisms. It is commercially available, but not widely used. Isotopically labeled compounds can be expensive to synthesize	Field application of SIP involves the deployment of ^{13}C-dioxane baited biotraps into groundwater wells. The baited biotraps will be withdrawn from the groundwater wells for laboratory analyses. The laboratory reports isotopically enriched contaminant into microbial cell structures (e.g., phospholipids, DNA) or generation of terminal products (e.g., CO_2) containing stable isotopes from the added ^{13}C-dioxane	Chiang et al., 2012

Exhibit 4. Continued

EMD	Description[a]	Application on 1,4-Dioxane	Reference
EAP	Transformation of surrogate compounds (probes) resembling contaminants produces a fluorescent (or other distinct) signal in cells which is then detected using a microscope. This provides direct evidence of enzyme activity and thus indirect evidence of biodegradation of contaminants	Field application of EAP involves the groundwater samples collected from 1,4-dioxane contaminated wells for analysis of EAPs, such as trans-cinnamonitrile, and 3-hydroxyphenylacetlyene (3-HPA)	Data not published
CSIA	CSIA is an analytical method that measures the ratios of naturally occurring stable isotopes in environmental samples. CSIA can be used to gain information relevant to potential contaminant sources, extent of degradation, and comingling of contaminant plumes relevant to environmental remediation decision makers	Both carbon and hydrogen CSIA methods for low-concentration levels of dioxane have been developed. CSIA is developed to gain relevant information on potential sources, extent of degradation and possibly mechanisms of degradation. Although CSIA methods are developed and some CSIA data have been collected, data interpretations have not yet been reported. Enrichment factors needed to estimate degradation mechanisms have not been established	CSIA methods and case studies are published in this special dioxane issue

EMD = environmental molecular diagnostics; qPCR = quantitative polymerase chain reaction; RT-qPCR = reverse transcriptase qPCR; CSIA = compound-specific isotope analysis; SIP = stable isotope probing; EAP = enzyme activity probes.

[a] ITRC Technical and Regulatory Guidance for Environmental Molecular Diagnostics (ITRC, 2013).

oxidizing bacteria (MOB) as a primary food source for cometabolic biodegradation of dioxane (still under study). Treatment train approaches for CVOCs and dioxane have been previously proposed; however, such approaches are still being researched.

As of today, technologies proven for treating large dilute dioxane plumes are limited. Groundwater pump-and-treat involving groundwater extraction for hydraulic control and *ex situ* treatment using advanced oxidation processes (AOP) or proprietary sorption materials are the most proven technologies for achieving low dioxane cleanup criteria.

U.S. DoD PERSPECTIVE OF DIOXANE ISSUES

The U.S. DoD has been addressing legacy environmental contamination for decades. While much progress has been made to date, emerging contaminants have the potential to delay site closure and other restoration goals. Dioxane is especially relevant to the DoD given widespread groundwater contamination at installations due to legacy use of chlorinated solvents. Specifically, TCE and TCA have been the most heavily used chlorinated solvents and typically co-occur in groundwater plumes due to their sequential

Exhibit 5. Review of *in situ* 1,4-dioxane treatment technologies

Process Options	**Description**	**Effectiveness for Dioxane**	**Implementability**	**Cost**
Air Sparging/SVE	Air Sparging involves the injection of air or oxygen into a contaminated aquifer to create an underground air stripper effect that removes volatile and semivolatile organic contaminants by volatilization. As the contaminant-laden air bubbles rise to the surface of the groundwater and into the vadose zone, the vapors are drawn off and treated by an SVE system	Low	Not effective for dioxane	Capital: High O&M: Moderate
In-Well Air Stripping	For in-well air stripping, air is injected into a double-screened well, causing the contaminants in the contaminated groundwater to transfer from the dissolved phase to the vapor phase in air bubbles. As the air bubbles rise to the surface of the groundwater, the vapors are drawn off and treated by a SVE system	Low	Not effective for dioxane	Capital: High O&M: Moderate
Aerobic Metabolic Bioremediation	Aerobic metabolic bioremediation involves addition of oxygen and bacterial culture to enhance the degradation of dioxane via metabolic microbial activity (in which the bacteria use contaminants as an energy/food source) under aerobic conditions	Likely moderate to high if relevant bacterial strains are present. Limited pilot study results have been documented	High/moderate for treating dioxane at localized hot-spot areas or in a permeable reactive barrier requiring a moderate number of injection points. Low for treating contaminants over large areas requiring many injection points, especially in off-site areas where accessibility may be an issue Pilot testing would be required	Capital: High/Moderate O&M: Moderate/Low

Exhibit 5. Continued

Process Options	Description	Effectiveness for Dioxane	Implementability	Cost
Aerobic Cometabolic	Aerobic cometabolic bioremediation involves addition of oxygen and a carbon substrate (e.g., alkane gases such as butane, propane, or methane) to enhance microbial breakdown of a contaminant, wherein the contaminant degrades incidentally by an enzyme or cofactor produced during microbial metabolism of the added carbon substrate. The oxygen and the alkane gas can be added via infusion (e.g., using iSOC® units) or via sparging. Bioaugmentation (addition of select bacteria) can be used to boost indigenous bacterial populations	Moderate to high	High/moderate for treating dioxane at localized hot-spot areas or in a permeable reactive barrier requiring a moderate number of injection points Low for treating dioxane over large areas requiring many injection points, especially in off-site areas where accessibility may be an issue Pilot-scale propane biostimulation has been demonstrated	Capital: High/Moderate O&M: Moderate/Low
Anaerobic Bioremediation	Anaerobic bioremediation involves addition of an electron donor substrate, such as hydrogen or a source of hydrogen, to reduce contaminant through anaerobic reductive reactions	Low	Not effective for dioxane	Capital: High/Moderate O&M: Moderate/Low
Chemical Reduction	Reducing chemicals, such as zero valent iron (ZVI), are used to chemically reduce the contaminants to nontoxic products. Chemicals can be injected via injection wells or into trenches	Low	Not effective for dioxane	Capital: High/Moderate O&M: Moderate/Low
Chemical Oxidation—Potassium Permanganate	$KMnO_4$ added to oxidize dioxane in the groundwater to carbon dioxide, manganese dioxide, and hydrogen	Low	Not effective for dioxane	Capital: Moderate O&M: Moderate

Exhibit 5. Continued

Process Options	Description	Effectiveness for Dioxane	Implementability	Cost
Chemical Oxidation—Activated Sodium Persulfate	Activated sodium persulfate added to oxidize dioxane in the groundwater to carbon dioxide, sodium, hydrogen, and sulfate.	High to Moderate	High/moderate—for treating dioxane at localized hot-spot or source areas, a moderate number of injection points and numerous injection events Low—for treating or containing contaminants over large areas requiring many injection points or in off-site areas where accessibility may be an issue. Also, persulfate longevity in the subsurface is on the order of only one to three months, typically requiring several re-injections thus reducing implementability	Capital: High O&M: Moderate
Chemical Oxidation—Ozone & Peroxide	Ozone and peroxide added to oxidize dioxane in the groundwater to carbon dioxide, oxygen, and water	High to Moderate	Moderate—for treating dioxane at localized hot-spot or source areas Low—for treating or containing dioxane over large areas requiring many injection wells or in off-site areas where accessibility may be an issue. A continuous ozone and peroxide delivery system and associated O&M would be required	Capital: High O&M: High
Chemical Oxidation—Fenton's Reagent	Hydrogen peroxide added with an iron catalyst to oxidize dioxane in the groundwater to water and oxygen	Moderate	Low/moderate - for treating dioxane at localized hot-spot or source areas requiring a moderate number of injection points Low—for treating or containing contaminants over large areas requiring many injection points or off-site, where accessibility may be an issue. Exothermic reactions produce high heat resulting in potential health and safety concerns. The longevity of reactions is on the order of only hours; therefore, multiple field injections are likely required to achieve cleanup goals. Treatability testing would be required	Capital: High O&M: Moderate to High

Exhibit 5. Continued

Process Options	Description	Effectiveness for Dioxane	Implementability	Cost
Monitored Natural Attenuation (MNA)	MNA is the reliance on natural attenuation processes to achieve site-specific remediation objectives within a reasonable time frame	Subject to site conditions	Moderate—a monitoring well network for tracking dioxane plume stability is typically not in place, additional wells and multiple years of monitoring may be required Critical parameters governing biodegradation are still under development to confirm the biodegradation mechanisms	Capital: Low O&M: Low
Phyto-remediation	Phytoremediation uses plants to uptake, transfer, stabilize, or destroy dioxane in groundwater	Moderate	Low for water table below 10 feet below ground surface (bgs) Moderate to high for shallow water table allowing plant roots to capture dioxane in groundwater May go deeper (>10 ft bgs) with modified tree-well system	Capital: Low O&M: Low

use prior to pollution prevention regulations (Mohr et al., 2010). Exhibit 7 proportionally illustrates the historic prevalence and co-occurrence of TCE, TCA, and 1,1-DCE (mutual daughter product of both TCE [hydrogenolysis] and TCA [dehydrohalogenation]) in groundwater across the portfolio of U.S. Air Force (USAF) sites. Exhibit 8 illustrates the corresponding co-occurrence of all USAF dioxane detections to date (>1,800 monitoring wells across 46 installations). TCA is clearly the source of the vast majority of dioxane observed at these sites. Additionally, some fraction may have originated from TCE particularly because TCA and its daughter products simply do not account for all dioxane detections in wells with TCE (Exhibit 8), although fate and transport differences as well as the sequential use of TCE and TCA complicate the interpretation of dioxane occurrence (Adamson et al., 2014). Further evidence is provided by the U.S. patent literature, which suggests some TCE formulations may have contained dioxane (Morrison & Murphy, 2015) and previous analyses of these data demonstrating categorical and quantitative statistical associations between TCE and dioxane (Anderson et al., 2012). Regardless, TCE and/or TCA account for essentially all dioxane contamination at industrial sites impacted by chlorinated solvents as shown in these data (Exhibit 8; Anderson et al.,2012) and elsewhere (Adamson et al., 2014).

Given the above, the USAF has adopted programmatic measures to facilitate the characterization of dioxane at TCE/TCA-impacted groundwater sites. However, the full scale of dioxane occurrence remains uncertain due to the lack of dioxane data at the majority of the sites identified in Exhibit 7. This situation is not unique to the USAF and is representative of the industry at large (Adamson et al., 2014). Moreover, evolving criteria among select regulatory authorities have rendered moot some early investigations that

Exhibit 6. Review of *ex situ* 1,4-dioxane pump-and-treat technologies

Process Options	Description	Effectiveness for Dioxane	Implementability	Cost
Adsorption—Granular Activated Carbon (GAC)	In adsorption, extracted groundwater is passed through treatment vessels containing GAC to concentrate contaminants on the surface of a carbon sorbent, which has to be regenerated or replaced periodically	Low	Not effective for dioxane	Capital: High O&M: Low/Moderate
Adsorption—AMBERSORB™ 563 Polymeric Adsorbent	Extracted groundwater is passed through a column containing a synthetic medium AMBERSORB™ 563, which has to be regenerated or replaced periodically	High	Moderate AMBERSORB™ 563 is a more recent proprietary product and currently has flow limitations	Capital: High O&M: High
Air Stripping	Air stripping partitions volatile organics from extracted groundwater by increasing the surface area of the contaminated water exposed to air. Aeration methods include packed towers, diffused aeration, tray aeration, and spray aeration	Low	Not effective for dioxane	Capital: High O&M: Low/Moderate
Chemical Oxidation—Ozone & Peroxide	Ozone & peroxide are used to create hydroxyl radicals, which break down organic contaminants into carbon dioxide & water	High	High (HiPOx system commercially available)	Capital: High O&M: High
Vertical Engineering Barriers (VEB)—Slurry wall, grout curtain, or cutoff wall	Vertical engineered barriers (VEBs) are subsurface barriers made of an impermeable material designed to contain or divert groundwater	Low: Dioxane is contained without treatment	Low when aquifer too deep or area is too large	Capital: High/Moderate depending on VEB size O&M: Low
Hydraulic barriers—Vertical or Horizontal Extraction Wells	Extraction wells are placed in strategic locations to hydraulically control migration of impacted groundwater and to remove contaminants	High	Moderate depending on capture zone and draw down. Horizontal wells may be more appropriate for off-site locations due to accessibility issues	Capital: High/Moderate O&M: Low
Permeable Reactive Barriers (PRB)—Injection point or injection wells installed perpendicular to the flow path to intercept groundwater flow	*In situ* permeable barrier installed across the flow path of the contaminated groundwater plume, allowing the water to flow through the barrier while reducing the concentration of contaminants by employing *in situ* remediation (e.g., bioremediation or chemical oxidation) within the barrier	Depends on selected approach	Low to moderate – depending on depth and length of barrier which may require numerous injection points and numerous injection events	Capital: High/Moderate O&M: Low/Moderate

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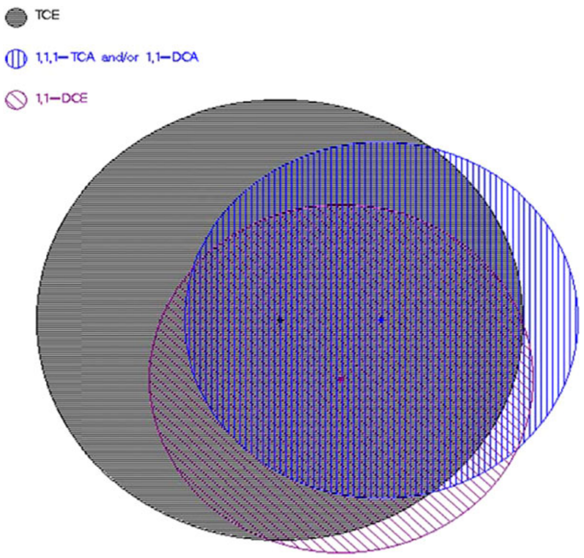

Exhibit 7. Three-way proportional Venn diagram illustrating the historic prevalence (i.e., site count) and co-occurrence of TCE, 1,1,1-TCA (and/or 1,1-DCA), and 1,1-DCE in groundwater across the portfolio of USAF sites

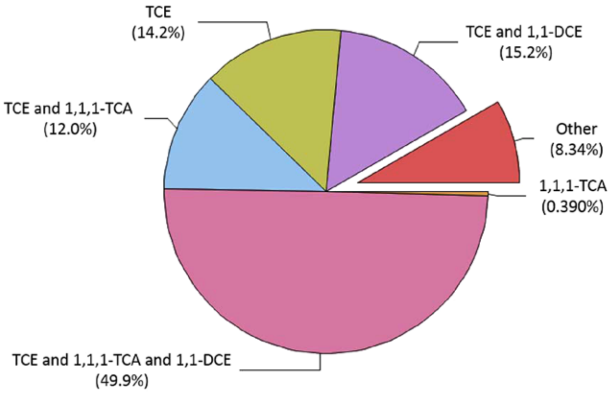

Exhibit 8. Co-occurrence of all 1,4-dioxane detections with 1,1,1-TCA (and/or 1,1-DCA) and TCE and 1,1-DCE from temporary and permanent monitoring wells among USAF installations. Data reflect all detections including "J" flagged and estimated values

screened dioxane from further consideration. Needless to say, much additional data are needed before the full extent of dioxane occurrence is fully quantified.

Notwithstanding the inconsistent regulations (or altogether lack thereof), universal consideration of dioxane occurrence is stymied in part due to specialized analytical methods beyond those traditionally used to investigate and monitor chlorinated solvents (e.g., USEPA methods 8260/8270) for accurate quantitation. Nevertheless, analytical limits of quantitation (LOQ) have greatly improved over the last decade (e.g., Park et al., 2005; Sun et al., 2005). Perhaps unsurprisingly, there is an inverse relationship evident between the mean LOQ and the frequency of detection (FOD) among USAF dioxane records over time (Exhibit 9). Because relatively few dioxane plumes (mostly those at high

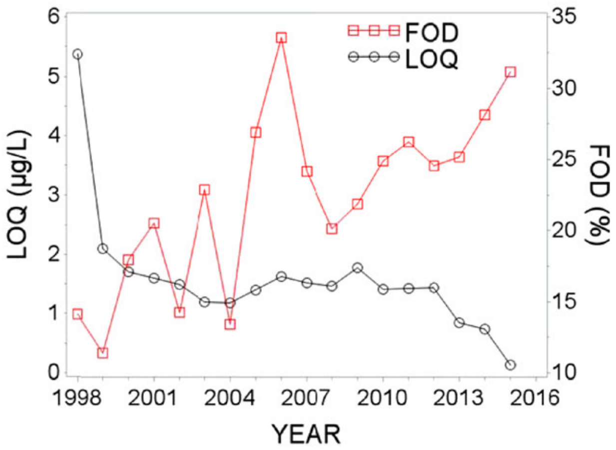

Exhibit 9. Temporal trends in the mean limit of quantification (LOQ) and frequency of detection (FOD) for 1,4-dioxane among approximately 30,000 groundwater samples collected to date from USAF installations

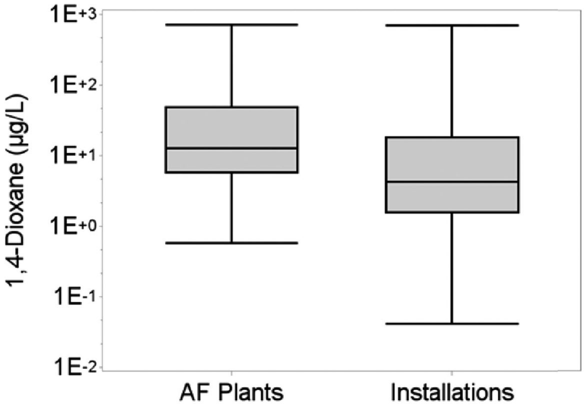

Exhibit 10. Distribution of historic maximum 1,4-dioxane groundwater concentrations from temporary and permanent monitoring wells among USAF installations ("AF Plants" refer to industrial complexes where historic aircraft manufacturing took place). Data only reflect valid quantitations (i.e., all "J" flagged and estimated values are omitted). The horizontal line represents the median, the gray box represents the inter-quartile range, and the upper and lower whiskers represent 95th percentiles

profile sites) have been (or are being) delineated, the increasing FOD trend mostly reflects discovery of diffuse dioxane plumes as confirmation sampling efforts progress across the portfolio of TCE/TCA-impacted groundwater sites. Exhibit 10 illustrates the distribution of observed concentrations at Air Force plants, where historic aircraft manufacturing took place, and other USAF installations. Although there is likely a low bias in at least some fraction of these data given the traditional dependence on USEPA Method 8270, typical concentrations are in the μg/L range indicative of chlorinated solvent sources (Exhibit 10).

Meanwhile, the DoD has invested heavily in the research and development of advanced characterization tools and remedial technologies to address dioxane contamination. Competitive funding initiatives, including the Strategic Environmental Research and Develop Program (SERDP) and the Environmental Security Technology Certification Program (ESTCP), have been instrumental to the current state of the science. To date, the DoD has invested millions of dollars in research and development of dioxane characterization and remediation. In particular, development of novel molecular biomarkers indicative of dioxane degradation (e.g., Gedalanga et al., 2014; Li et al., 2013) and mechanistic understanding of intrinsic degradation processes have led to the growing awareness that dioxane can be effectively remedied by natural attenuation at some sites (Adamson et al., 2015).

In summary, the dioxane problem at DoD sites is defined by a yet unknown scale of occurrence, on-going analytical evolution, and regulatory inconsistency. Until these issues are definitively addressed, further progress toward universally addressing dioxane across the portfolio of DoD sites is unlikely. Doing so will require multilateral stakeholder engagement on a site-specific basis. Overall, the DoD's dioxane problem exemplifies the challenge with emerging contaminants and the role they currently play at contaminated sites.

SUMMARY AND FUTURE PERSPECTIVES

With the apparent decreasing trend of state regulatory criteria for dioxane, reliable analytical methods are needed. This article demonstrates that inconsistent analytical results are obtained between the two most common analytical methods for dioxane. Significant bias has also been identified between different laboratories. The lack of dioxane recovery correction with isotope dilution during a laboratory's sample preparation can lead to significantly lower reporting concentrations. This suggests an urgent need of developing reliable sample preparation procedures and analytical methods.

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Dioxane is miscible in water and migrates with limited retardation in the aquifer. Therefore, large and dilute dioxane footprints are expected based on its physicochemical characteristics. Recently, it has been documented that dioxane plumes are not in fact always advancing ahead of CVOC plumes. An increasing number of case studies have shown dioxane natural attenuation is possible. For many complex CVOC sites, the path to site closure is already uncertain. If cost-effective MNA and biostimulation of dioxane can be considered as part of the site remedial strategy, the site managers can continue to focus on remediating the primary contaminants of concern. In the meantime, more case studies are also needed to identify the favorable site conditions for natural attenuation to occur and when and how *in-situ* biostimulation/bioaugmentation can be implemented with confidence of success.

In recent years, the remediation industry has developed a better understanding of dioxane behaviors, natural attenuation potential, and active treatment technology options for dioxane. However, reliable analytical methods, more real-world case studies on *in situ* remediation and natural attenuation, and dioxane plume management strategies are still needed. In summary, the authors recommend the remediation practitioners to collaborate and fill in the following knowledge gaps:

- Proper and consistent standard methods to improve dioxane data quality;
- Case studies assessing and demonstrating dioxane plume stability;

- High-resolution site characterization to improve understanding of dioxane mass storage and discharge;
- Geochemical indicators to characterize attenuation of dioxane;
- Usefulness and applicability of EMDs for evaluating dioxane biodegradation;
- Cost and technology benefits of treating CVOCs and dioxane concurrently or sequentially;
- Green and sustainable aspects of dioxane plume management;
- Low-cost *ex situ* treatment technologies;
- Full-scale demonstration of in situ dioxane biodegradation; and
- Technical guidance on feasibility of MNA for dioxane.

The techniques available today to fill in the knowledge gaps have become more powerful, specific, and effective comparing to the era of developing early technologies for treating petroleum compounds or chlorinated solvents. High-resolution site characterization, molecular diagnostic tools and compound-specific isotopic tools have changed the ways for researchers to confirm degradation mechanisms and for practitioners to optimize treatment systems. This article summarizes the current tools and technologies available for dioxane characterization and remediation. We anticipate smart site characterization and combined treatment technologies to reduce social, economic, and environmental risks associated with the releases of dioxane will lead the way for managing dioxane plumes.

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Sheau-Yun (Dora) Chiang, PhD, P.E., is director of emerging contaminants at AECOM. She holds a PhD from Georgia Institute of Technology and over 15 years of consulting experience in the areas of contaminated site investigation and remediation. She works collaboratively with senior management to assess and determine the firm's position in global contaminated site cleanup and closure.

Richard (Hunter) Anderson, PhD, is an environmental scientist at the U.S. Air Force Civil Engineer Center (AFCEC) in San Antonio, Texas. He provides technical expertise to AFCEC's Environmental Restoration Program in the areas of soil science, environmental toxicology, and quantitative data analyses and serves as the U.S. Air Force Restoration liaison to the U.S. DoD's Strategic Environmental Research and Development and Environmental Security Technology Certification Programs (SERDP/ESTCP). Dr. Anderson received his BS/MS in environmental science from Oklahoma State University and PhD in soil science from Ohio State University.

Michael Wilken is environmental analytical leader in the Environmental Remediation and Restauration Group at The Dow Chemical Company in Midland, Michigan. He received a diploma from the Technical University in Berlin, Germany. After being the manager of an environmental laboratory with the focus on dioxins and related components for 10 years, he had his own worldwide consultancy business for another nine years before he joined Dow and relocated to the United States 12 years ago. For the first seven years, he was leading the dioxin laboratory within the company and is since the liaison between the Analytical Chemistry and the Remediation Leader with the focus on the data quality.

Claudia Walecka-Hutchison, PhD, is an EH&S remediation manager at The Dow Chemical Company. In this role, she is involved in developing site-specific solutions for Dow's global remediation projects including maintaining active research studies. Claudia has over 10 years of experience in site investigation and remediation with extensive expertise working with chlorinated solvents and petroleum hydrocarbons. Claudia specializes in bioremediation and holds a PhD in soil, water, and environmental science and an MS in hydrology from the University of Arizona.