IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

#### BEFORE THE PATENT TRIAL AND APPEAL BOARD

#### PEROXYCHEM LLC Petitioner,

v.

### INNOVATIVE ENVIRONMENTAL TECHNOLOGIES, INC. Patent Owner.

U.S. Patent No. 7,531,709 to Michael Scalzi, et al. Issue Date: May 12, 2009 Title: Method For Accelerated Dechlorination Of Matter

Inter Partes Review No.: Unassigned

#### PETITION FOR INTER PARTES REVIEW OF U.S. PATENT NO.

7,531,709

#### **TABLE OF CONTENTS**

I.	INTRODUCTION		1
II.	<b>OVERVIEW</b> 1		
III.	MAN	NDATORY NOTICES UNDER 37 CFR § 42.8	6
	А.	Real party-in-interest	6
	B.	Related matters	6
IV.	GRC	OUNDS FOR STANDING	8
V.	SPECIFIC RELIEF REQUESTED		
VI.	OVE	RVIEW OF THE '709 PATENT	8
	A.	Summary of the '709 Patent	8
	B.	Prosecution History	11
	C.	Claim Construction	12
	D.	Priority Date of the Challenged Claims	14
	E.	Person of Ordinary Skill in the Art	14
	F.	The State of the Art and The Challenged Claims	15
VII.	THE	CHALLENGED CLAIMS ARE NOT PATENTABLE	20
	А.	Statutory Grounds for Challenges	20
	B.	Statement of Non-Redundancy of the Grounds.	21
	C.	Ground 1: Claims 1, 7, 9 and 14 are anticipated under 35 U.S.C. § 102(b) by Hitchens	23

Ground 2: Claims 1, 3, 7, 9 and 14-16 are anticipated under 35 U.S.C. § 102(e) by U.S. Patent Application Publication No. 2002/0151602	30
Ground 3: Claims 1, 3, 7 and 14 are anticipated under 35	
U.S.C. § 102(b) by Alvarez et al.	37
Ground 4: Claims 1-3, 5, 7, 11, 12 and 14-16 are rendered	
obvious under 35 U.S.C. § 103 by Rice in view of Cantrell	44
Ground 5: Claims 4 , 10 and 13 are unpatentable under 35	
U.S.C. § 103(a) over Vance in view of Schuring	54
Ground 6: Claim 8 is unpatentable under 35 U.S.C. § 103(a)	
over Hitchens in view of Grasso	57
Ground 7: Claim 6 is unpatentable under 35 U.S.C. § 103(a)	
over Hitchens in view of Odom	58
clusion	59
	<ul> <li>35 U.S.C. § 102(e) by U.S. Patent Application Publication No. 2002/0151602</li> <li>Ground 3: Claims 1, 3, 7 and 14 are anticipated under 35 U.S.C. § 102(b) by Alvarez et al.</li> <li>Ground 4: Claims 1-3, 5, 7, 11, 12 and 14-16 are rendered obvious under 35 U.S.C. § 103 by Rice in view of Cantrell.</li> <li>Ground 5: Claims 4, 10 and 13 are unpatentable under 35 U.S.C. § 103(a) over Vance in view of Schuring.</li> <li>Ground 6: Claim 8 is unpatentable under 35 U.S.C. § 103(a) over Hitchens in view of Grasso.</li> <li>Ground 7: Claim 6 is unpatentable under 35 U.S.C. § 103(a)</li> </ul>

#### **PETITIONER'S EXHIBIT LIST**

PXY-1001	U.S. Patent Number 7,531,709 (the "709 Patent")
PXY-1002	U.S. Patent Number 7,531,709 File History
PXY-1003	U.S. Patent Number 6,265,205 ("Hitchens")
PXY-1004	Publication WO 98/49106 ("Alvarez")
PXY-1005	TECHNOLOGY NEWS AND TRENDS, EPA newsletter publication. June 2002 ("TNT")
PXY-1006	U.S. Patent No. 6,001,252 ("Rice")
PXY-1007	U.S. Patent No. 5,789,649 ("Batchelor")
PXY-1008	U.S. App. No. 11/423,749 (the "749 Application")
PXY-1009	U.S. Provisional Patent App. No. 60/437,983 (the "'983 Provisional Application")
PXY-1010	U.S. Patent Application Publication No. 2002/0151602 ("Vance")
PXY-1011	Expert declaration of Dr. John A. Bergendahl
PXY-1012	Hazardous Materials, Elsevier Science Press B.V., Amsterdam, The Netherlands. "Geotechnical Techniques for the Construction of Reactive Barriers" March 1999 ("Day")
PXY-1013	Environmental Science & Technology, Vol. 31, No. 12, pp. 3448-3454, "DDT, DDD and DDE Dechlorination by Zero-Valent Iron," 1997 ("Sayles")
PXY-1014	Dominic Grasso, Hazardous Waste Site Remediation, Lewis Publishers, Boca Raton (1993) ("Grasso")
PXY-1015	U.S. Patent No. 6,596,190 ("Igawa")
PXY-1016	U.S. Patent No. 5,658,093 ("Kawabata")
PXY-1017	United States Environmental Protection Agency, "Reference Guide to Non-Combustion Technologies for Remediation of Persistent Organic Pollutants in Stockpiles and Soil" ("USEPA")

PXY-1018	Matheson and Tratnyek, "Reductive Dehalogenation of
	Chlorinated Methanes by Iron Metal," Environ. Sci.
	Technol., 1994, 28 (12), pp. 2045–2053 ("Matheson")
PXY-1019	Interstate Technology and Regulatory Cooperation Work
	Group, "Natural Attenuation of Chlorinated Solvents in
	Groundwater: Principles and Practices," September 1999
	("ITRCW")
PXY-1020	Evan K. Nyer, et al., In Situ Treatment Technology, 2d. Ed.,
	Lewis Publishers, Boca Raton (2001) ("Nyer")
PXY-1021	Bruce E. Rittman and Perry L. McCarty, Environmental
	Biotechnology: Principles and Applications, McGraw Hill,
	NY (2001) ("Pittman")
PXY-1022	Appendix A "Related Patents"
PXY-1023	U.S. Patent No. 7,129,388 (the "Parent Patent")
PXY-1024	U.S. Patent No. 5,032,042 ("Schuring")
PXY-1025	U.S. Patent No. 5,587,317 ("Odom")
PXY-1026	U.S. Patent No. 5,857,810 ("Cantrell")

#### I. INTRODUCTION

Pursuant to 35 U.S.C. § 311 et seq. and 37 CFR § 42.1 et seq., Peroxychem, LLC ("Petitioner") petitions for *Inter Partes* Review, seeking cancellation of Claims 1-16 (the "Challenged Claims") of U.S. Patent No. 7,531,709 to Scalzi *et al.* (the "709 Patent"), attached herein as Exhibit PXY-1001, which is owned by Innovative Environmental Technologies, Inc. ("IET" or "Patent Owner"). Petitioner respectfully submits that the Challenged Claims are unpatentable under 35 U.S.C. §§102 and/or 103 in view of the prior art references discussed herein. This Petition demonstrates by at least a preponderance of the evidence that there is a reasonable likelihood that Petitioner will prevail with respect to at least one of the Challenged Claims. Accordingly, it is respectfully requested that the Board institute an *inter partes* review of the '709 Patent pursuant to 37 CFR § 42.108.

#### II. OVERVIEW

The Challenged Claims are unpatentable as anticipated and/or obvious over the prior art. The alleged "inventions" are simply recitations of what was known and practiced in the art of in situ soil remediation and *at best* involve no more than the mere obvious *modification of prior art teachings*, with a reasonable expectation of success. As such, these claims fall squarely, and fatally, under *KSR Int'l Co. v. Teleflex Inc.*, 550 U.S. 398, 417 (2007), and should therefore be cancelled. At least as early as the 1990's, methods for addressing toxic pollutants in contaminated ground without removing the soil (in situ) were well known. The '709 Patent describes and claims a method for in situ soil remediation by supplying a first and a second remediation element into the soil so that naturally occurring processes dechlorinate/dehalogenate contaminants in the soil. However, methods for in situ soil remediation by supplying the first and/or the second remediation element into the soil were described individually, and in combination with each other, in prior art well before the 2003 priority date of the '709 Patent.

Specifically, the '709 Patent describes and claims a method for in situ soil remediation by supplying into the soil: (1) a zero valent metal, typically iron, for reductive dechlorination/dehalogenation of contaminants in the soil; and (2) an organic hydrogen donor to accelerate dechlorination/dehalogenation of the contaminants in the soil by indigenous anaerobic bacteria. However, methods for in situ soil remediation by supplying one or both of these remediation elements were in practice across the country at least as early as 1995, and routinely practiced prior to 2002 as described in Technology News and Trends ("TNT")(PXY-1005).

Moreover, by the late 1990's the advantageous symbiotic relationship between supplying both of these remediation elements was clearly understood. Several prior art references disclose supplying both a zero valent metal and an organic hydrogen donor into the soil for remediation.

DM2\6357536.1

2

For example, Vance et al. ("Vance") (Exhibit PXY-1010) describes that

"since the mid-1990's, there have been a series of dramatic developments for the in-situ treatment of chlorinated solvents, " (PXY-1010, ¶ [0002]) and describes a method "based on the sequential reduction of chlorinated hydrocarbons to innocuous end products." *Id.* Vance describes a dechlorination method of injecting zero valent iron along with a carbohydrate (organic hydrogen donor), specifically corn syrup, into the soil, as shown in Fig. 5. *Id.*, ¶ [0121], Fig. 5; PXY-1011, ¶ 87.



Likewise, both Hitchens *et al.* ("Hitchens")(Exhibit PXY-1003) and Alvarez *et al.* ("Alvarez") (Exhibit PXY-1004) describe benefits of supplying both a mixture of zero valent metal and an organic hydrogen donor for "enhancing the rate of biodegradation of chlorinated organic compounds." PXY-1003, 1:10-15; PXY-1011, ¶ 77. Specifically, Hitchens describes degrading chlorinated solvents with granular iron (zero valent iron) and introducing glucose, lactate or formates (organic hydrogen donors) into the soil. PXY-1011, ¶ 76. Significantly, Hitchens identifies the identical benefit of supplying both of these remediation elements that the '709 Patent describes, stating "this chemical degradation process is assisted by a parallel biological route where the growth of methogenic bacteria is stimulated by the produced hydrogen, and these organisms enzymatically dechlorinating additional contaminants." PXY-1003, 3:3:39-56; PXY-1011, ¶ 76.

Further, Alvarez describes reductive dechlorination by iron corrosion, which produces H<sub>2</sub> and, as the dechlorination method is anaerobic, it also "support[s] the growth of useful bacteria." PXY-1004, p. 7; PXY-1011, ¶ 96. Alvarez's methods "for bioremediation of mixed waste aquifers, [are] based on a synergistic combination of reductive treatment using zero-valent iron and anaerobic biotransformations." PXY-1004, Abstract; PXY-1011, ¶ 96. Importantly, Alvarez describes the metal composition may be advantageously augmented by introducing organic hydrogen donors to stimulate the natural reductive dechlorination process. PXY-1004, pp. 23, 24 and 58; PXY-1011, ¶ 96.

It is these very prior art methods that are recited in the Challenged Claims as shown in Claim 1:

1. A method for accelerated anaerobic dechlorination of subsoil matter, comprising the steps of:

*supplying a mixture including a zero valent metal into soil pathways to biologically react with the dissolved chlorinated solvents in the groundwater; and* 

supplying an organic hydrogen donor into the soil pathways to produce dechlorinating conditions such that indigenous anaerobic bacteria biodegrade residual concentrations of chlorinated solvents. (emphasis added) These two elements are found in the prior art in combination (Vance,

Hitchens, Alvarez, TNT, Day, Igawa, Cantrell ) and individually (Rice, Batchelor,

Sayles, Kawabata). PXY-1011, ¶ 47, Table 1.

'709 Patent Dechlorination Method		
Claim 1	Element (1):	Element (2):
	Supply Zero Valent Metal	Supply Organic Hydrogen Donor
	Disclosed examples:	Disclosed examples: Glucose,
	Zero Valent Iron (Fe <sup>(0)</sup> ),	Sucrose, Sugars, Alcohols, Chitin,
	Iron Powder	Propionates, Lactates, Acetates,
	PXY-1001, 5:41-59, 8:1	Butyrate
		PXY-1001, 4:29-32, 6:57-60
<b>Prior Art Refe</b>	rences	
TNT 2002	Zero Valent Iron (Fe <sup>(0)</sup> )	Organic Mulch, Organic Carbon
	<i>PXY-1005, pp. 2-3</i>	PXY-1005, pp. 1-2
Vance 2000	Zero Valent Iron (Fe <sup>(0)</sup> )	Corn Syrup (sugar)
	PXY-1010, ¶¶ [0118]-	<i>PXY-1010</i> , ¶¶ <i>[0118]-[0120]</i>
	[0120]	
Igawa 1999	Fine Iron Particles	Sucrose, Glucose
	PXY-1015, Abstract	PXY-1015, 5:10-21
Day 1999	Granular Iron	Guar Gum (sugar)
	<i>PXY-1012, p. 3</i>	PXY-1012, pp. 5-6
Hitchens 1998	Zero Valent Iron (Fe <sup>(0)</sup> )	Glucose, Acetate, Formate,
	PXY-1003, 3:39-56	Methanol (Alcohol), Lactate,
		Propionate, Butyrate
		PXY-1003, 2:32-44
Rice 1998		Lactate, Propionate And Acetate
		PXY-1006, 4:9-10
Alvarez 1997	Zero Valent Iron (Fe <sup>(0)</sup> )	Methanol (Alcohol), Acetate
	<i>PXY-1004,</i> ¶¶ <i>10, 17</i>	<i>PXY-1004, pp. 23-24, 26</i>
Cantrell 1997	Zero Valent Iron (Fe <sup>(0)</sup> )	Guar Gum (sugar)
	<i>PXY-1026, 4:27-35,5:33-35</i>	<i>PXY-1026, 3:54-60</i>
Sayles 1997	Zero Valent Iron (Fe <sup>(0)</sup> )	
	<i>PXY-1013, p. 3448</i>	
Batchelor	Zero Valent Iron (Fe <sup>(0)</sup> )	
1995	PXY-1007, 4:41-47, claim 1	
Kawabata		Sugar, Chitin

1994	PXY-1016, 6:14-20

Claiming the state of the art is *not* inventive and combining known methodologies for their intended purpose is *not* an innovation. Dependent Claims 2-16 also represent no more than what was known and routine practice in the art, for example using injection rods (*Claim 3*), using colloidal suspensions (*Claims 11*, *15 and 16*), and cleansing lines after use (*Claim 13*). The claims of the '709 Patent are known as are their functions and predictable results. PXY-10011, ¶ 10. A person of ordinary skill in the art ("POSA") would have readily known and undertaken these steps in the remediation of contaminated soil and would further have performed these simple combinations if not previously known because the purposes of these remediation methodologies were well known in the art and the results of the combinations were predictable in view of that knowledge. PXY-1011, ¶ 10.

#### III. MANDATORY NOTICES UNDER 37 CFR § 42.8

#### A. Real party-in-interest

The real parties-in-interest in this Petition are Peroxychem LLC ("Petitioner"), Scott Steffl, and Bruce Lerner.

#### **B.** Related matters

#### 1. Judicial Matters

As of the filing date of this Petition and to the best knowledge of the

Petitioner, the '709 Patent is involved in the following litigation:

Innovative Environmental Technologies, et al. v. Peroxychem LLC, Scott

Steffel and Bruce Lerner, Eastern District of Pennsylvania, Case No. 15-3145,

filed on June 5, 2015.

*Peroxychem LLC v. Innovative Environmental Technologies, et al.*, Eastern District of Pennsylvania, Case No. 14–6446, filed on November 17, 2014.

#### 2. Related Patents

See Appendix A attached herein as Exhibit PXY-1022.

#### 3. Lead/Back-up Counsel and Service information:

Lead Counsel: D. Joseph English, USPTO Reg. No. 42,514 DUANE MORRIS LLP, 505 9th St. NW, Suite 1000, Washington, D.C. 20004 P: (202) 776-5228; F: (202) 776-7801; djenglish@duanemorris.com

Back-Up Counsel: Patrick C. Muldoon, USPTO Reg. No. 47,343
DUANE MORRIS LLP, 505 9th St. NW, Suite 1000, Washington, D.C. 20004
P: (202) 776-7840; F: (202) 776-7801; pcmuldoon@duanemorris.com
Samuel W. Apicelli, USPTO Reg. No. 36,427,
DUANE MORRIS LLP, 30 South 17<sup>th</sup> Street, Philadelphia, PA 19103

P: (215) 979-1255; F: (215) 979-1020; swapicelli@duanemorris.com

#### **IV. GROUNDS FOR STANDING**

Petitioner certifies that the Patent for which review is sought is available for *inter partes* review and that Petitioner is not barred or estopped from requesting an *inter partes* review challenging the patented claims on the grounds identified in this Petition. 37 CFR § 42.104(a).

#### V. SPECIFIC RELIEF REQUESTED

Petitioner asks that the Board review the accompanying prior art and analysis and institute an *inter partes* review of the Challenged Claims of the '709 Patent pursuant to 37 CFR § 42.108 and cancel those claims as unpatentable.

#### VI. OVERVIEW OF THE '709 PATENT

#### A. Summary of the '709 Patent

The '709 Patent is entitled "METHOD FOR ACCELERATED DECHLORINATION OF MATTER" and issued May 12, 2009, from an application filed June 13, 2006.

The '709 Patent discloses that it "relates to accelerated dechlorination of subsurface matter by anaerobic microorganisms in conjunction with oxygen scavengers, vitamins, nutrients, and zero valent metals." PXY-1001, 1:18-21. The '709 Patent acknowledges that it was well known that certain anaerobic microorganisms have the capability to decompose a wide range of highly chlorinated compounds and that the use of such anaerobic microorganisms was well known for in situ treatment of subsoil matter contaminated with chlorinated compounds. PXY-1001, 1:64-67. The '709 Patent further discloses that such known decontamination methods were disadvantageous due to the slow growth of anaerobic microorganisms in situ, particularly when compared to aerobic organisms. PXY-1001, 2:1-7.

The '709 Patent then touts as the alleged invention a soil remediation process that achieves accelerated dechlorination of subsoil matter by stimulating anaerobic microorganisms to increase the rate of biological mineralization of the solvents. PXY-1001, 2:11-14. The disclosed soil remediation process consists of a colloidal suspension of metal powder, organic hydrogen donor, chemical oxygen scavengers, nutrients, and vitamin stimulants which is delivered to the subsoil matter to be remediated by a closed system. PXY-1001, 2:14-24. The figure of the '709 Patent illustrates a preferred delivery system. PXY-1001, 4:46-47.

The '709 Patent discloses a single embodiment that includes the following steps:

Step 1: Subsurface Pathway Development ...

Step 2: Sodium Sulfite Nutrient and Micro Nutrient Injection...

Step 3: Zero Valent Metal Injection ...

Step 4: Anaerobic Hydrogen Source Injection...

Step 5: Sodium Sulfite/Nutrient Injection...

Step 6: Post Liquid Injection--Gas Injection...

#### PXY-1001, 2:50 - 3:42.

The '709 Patent discloses that the metal powder is a zero valent metal which may be zero valent iron. PXY-1001, 4:14-16, Claim 14. The '709 Patent recognizes that zero valent metals were known to have "good reducing power" to rapidly reduce high concentrations of dissolved phase chlorinated solvents when delivered to the contaminated subsoil matter. PXY-1001, 4:16-27.

The '709 Patent discloses that the organic hydrogen donor is an electron donor source to provide hydrogen such as "a polylactate ester, glycerol tripolylactate, xylitol pentapolylactate, or sorbitol hexapolylactate, lactates, acetate, propionates, sugars, glucose, etc." PXY-1001, 4:30-33. The '709 Patent also discloses organic hydrogen donors such as "glucose, sucrose, alcohols, propionates, lactates, acetates, chitin, polylactate esters, glycerol tripolylactate, xylitol pentapolylactate, and sorbitol hexapolylactate." PXY-1001, 2:14-19. The '709 Patent recognizes that the organic hydrogen donor, when delivered to the contaminated subsoil matter, will be cometabolized by indigenous anaerobic bacteria to produce the conditions necessary for the bacteria to biodegrade chlorinated solvents found in the subsoil matter. PXY-1001, 4:33-36.

The '709 Patent discloses that the nutrients delivered to the subsoil matter may include nitrogen, organic ammonia, and ortho-phosphate. PXY-1001, 2:64-65, 4:10-13.

The Challenged Claims<sup>1</sup> are directed to a method for accelerated anaerobic dechlorination of subsoil matter by supplying into soil pathways: (1) zero valent metal; and (2) an organic hydrogen donor.

#### **B. Prosecution History**

The '709 Patent issued on May 12, 2009, from U.S. Application No. 11/423,749 filed on June 13, 2006 (the "'749 Application")(Exhibit PXY-1008) which claims priority of U.S. Application No. 10/610,558 filed July 2, 2003, now U.S. Patent No. 7,129,388 (the "Parent Patent")(Exhibit PXY-1023), with each claiming priority to U.S. Provisional Application No. 60/437,983 filed January 6, 2003.

A copy of the U.S. Patent and Trademark Office file for the '709 Patent is attached herein as Exhibit PXY-1002. The only rejection issued by the examiner

<sup>&</sup>lt;sup>1</sup> For the purposes of this Petition, Petitioner assumes, without conceding, that the claim language "to biologically react with the dissolved chlorinated solvents in the ground water" and "to produce dechlorinating conditions such that indigenous anaerobic bacteria biodegrade residual concentrations of chlorinated solvents" is granted patentable weight under *Biodelivery Sciences Int'l, Inc. v. RB Pharm., Inc.*, Case IPR2014-00325, Paper 43, 6 (PTAB June 30, 2015).

during the prosecution in the '709 Patent was a nonstatutory double patenting rejection over Claims 1-10 of the Parent Patent.

#### C. Claim Construction

Under 37 CFR § 42.100(b), the terms of the claims of the '709 Patent are to be given their broadest reasonable interpretation in light of the specification of this Patent. Consistent with this standard, a proposed interpretation for certain claim terms is provided below. It is noted that this interpretation is only applicable to the *inter partes* review sought herein and should not be construed as constituting, in whole or in part, the Petitioner's own interpretation of any claims for any other purposes, including any litigation. Accordingly, Petitioner expressly reserves the right to present an interpretation of a claim term in other proceedings, which is different, in whole or in part, of that presented in this Petition.

#### 1. Proposed construction of "organic hydrogen donor"

The term "organic hydrogen donor" is recited in the only independent claim e.g., supplying an organic hydrogen donor into the soil pathways." PXY-1001, Claim 1. While the term "organic hydrogen donor" is not a term of art and not expressly defined in the specification, it is used extensively in the '709 Patent to refer to several examples of organic hydrogen donors, specifically glucose, sucrose, sugars, alcohols, propionates, lactates, acetates, chitin, butyrate, poly lactate esters, glycerol tripolylactate, xylitol pentapolylactate, and sorbitol hexapolylactate. PXY-1011, ¶ 73.

The invention also confirms that zero valent metal, when added with other compounds which provide a source of electron donors, nutrients, and reducing agents, will stimulate naturally occurring microorganisms. PXY-1001, 6:15-18.

[O]rganic hydrogen donor such as glucose, sucrose, alcohols, propionates, lactates, acetates, chitin, poly lactate esters, glycerol tripolylactate, xylitol pentapolylactate, and sorbitol hexapolylactate. Exhibit PXY-1001, 2:16-19.

With the addition of an electron donor source to provide hydrogen, the biodegradation process is initiated. An organic hydrogen donor such as a poly lactate ester, glycerol tripolylactate, xylitol pentapolylactate, or sorbitol hexapolylactate, lactates, acetate, propionates, sugars, glucose, etc. is now injected with the intent of being cometabolized by indigenous anaerobic bacteria to produce dechlorinating conditions necessary for indigenous anaerobic bacteria to biodegrade residual concentrations of chlorinated solvents. Exhibit PXY-1001, 4:28-36.

9. The method of claim 1 wherein said organic hydrogen donor is from the group consisting of lactate, propionate, chitin, butyrate, acetate, sugars, glycerol tripolylactate, xylitol pentapolylactate, and sorbitol hexapolylactate. PXY-1001, Claim 9

Thus a POSA would understand the term "*organic hydrogen donor*" as used in the passages above, under the broadest reasonable interpretation to mean "*a*  biodegradable organic compound capable of producing hydrogen through microbiological activity." PXY-1011, ¶ 74.

#### D. Priority Date of the Challenged Claims

Each of the Challenged Claims is directed to a method of soil remediation. PXY-1001, Claims 1-16. The earliest priority claim in the '709 Patent is to U.S. Provisional Patent App. No. 60/437,983 filed January 6, 2003 (the "'983 Provisional Application"), attached herein as Exhibit PXY-1009. For the purposes of this Petition, Petitioner does not challenge the claim of priority.

#### E. Person of Ordinary Skill in the Art

A Person of Ordinary Skill in the Art ("POSA") is a hypothetical person who is presumed to be aware of all pertinent prior art, thinks along conventional wisdom in the art, and is a person of ordinary creativity. With respect to the '709 Patent, a POSA would have had education and/or experience in the field of environmental science, engineering (civil, environmental or chemical), for the same, and knowledge of the scientific literature concerning the same, specifically in the area of soil and groundwater remediation. The education and experience levels may vary between persons of ordinary skill, with some persons holding a Bachelor's degree with 4-5 years of relevant work experience, or others holding more advanced degrees—e.g., Masters or Ph.D.—but with fewer years of experience. PXY-1011, ¶ 23. A POSA may work as part of a multi-disciplinary team and draw upon not only his or her own skills, but also take advantage of certain specialized skills of others on the team to solve a given problem. *Id.* at  $\P$  24.

#### F. The State of the Art and The Challenged Claims

As of January 2003, the purported invention of the '709 Patent was known in the state of the art. More specifically, it was known in the art to dechlorinate contaminated soil using zero valent metals and organic hydrogen donors, either alone or in combination. Further, the use of injection wells to treat contaminated soil and groundwater had long been known and practiced in the art as shown in the Figure 3.2 below from Grasso:



PXY-1014, J-12; PXY-1011, ¶¶ 44-46.

#### 1. The use of zero valent metals was known in the art

Dechlorination (or dehalogenation) of contaminants in the soil and ground water was accomplished prior to January 2003 through abiotic reduction with an elemental metal. PXY-1020, 467-470; PXY-1021, 719; PXY-1011, ¶ 48. Batchelor et al. ("Batchelor") describes the treatment of contaminated soil with the introduction of elemental metals (zero valent iron), and observes that using iron for the reduction of chlorinated solvents is a known and successful method. PXY-1007, 1:49-64, 2:1-10; PXY-1011, ¶ 49.

As early as 1994, studies confirmed the various pathways for a zero valent metal, such as iron, to effect (A) Direct Reduction at the Metal Surface Fe<sup>2+</sup> dechlorination. Matheson et al. ("Matheson")(PXY-1018) describe three RH + CIdechlorination pathways: (A) direct (B) Reduction by Ferrous Iron  $RX + H^+$ reduction, wherein the iron supplies an ►Fe<sup>2+</sup> RH + CIelectron directly to the chlorinated H<sub>2</sub>O compound; (B) reduction by ferrous iron OH - + H 2 (i.e. iron in aqueous solution), in which the (C) Reduction by Hydrogen with Catalysis ► Fe 2+ solubilized iron provides an electron to the H<sub>2</sub>0 chlorinated compound; and (C) reduction RX - + H 2 by hydrogen, wherein hydrogen produced  $RH + CI^{-} + H^{+}$ 

16

catalyst

as a result of anaerobic corrosion of iron performs dechlorination. PXY-1018, 2046; PXY-1011, ¶¶ 50-51.

Not only was the use of zero valent metals described in the relevant literature prior to January 2003, such use was in practice across the country. For example, in July 2000 NASA completed a pilot-scale field test of a zero valent iron process to address trichloroethene contamination in Huntsville, Alabama. PXY-1005, p. 3; PXY-1011, ¶ 52.

#### 2. The use of organic hydrogen donors was known in the art

Dechlorination accomplished by the introduction of an "electron donor" (organic hydrogen donor) to encourage indigenous anaerobic bacteria to decompose the chlorinated compounds was known prior to January 2003. PXY-1011, ¶ 53.

The biodegradation of chlorinated solvents was well understood at the time of the '709 Patent. Processes through which chlorinated compounds could biodegrade include: (1) direct oxidation, wherein the chlorinated compound is used as an electron donor and broken into inorganic molecules; (2) reductive dehalogenation (dechlorination), wherein chlorine ions are replaced with hydrogen ions; and (3) co-metabolism, wherein the metabolizing of a carbon compound results in the conversion of proximate chlorinated compounds to another chemical. PXY-1019, 8; PXY-1011, ¶ 54. It was further known to use organic hydrogen donors to accelerate these biodegradation processes. PXY-1020, 358-59; PXY-1021, 718-19; PXY-1011, ¶ 55; *see also* PXY-1014, J-26-27; PXY-1020, 334-36, 340-45, 357-61. For example, Rice et al. ("Rice")(Exhibit PXY-1006) describes "a method for in situ anaerobic dehalogenation of a halogenated organic compound in a ground water plume" in which a deoxygenated aqueous solution including an organic hydrogen donor (lactate, acetate or formate) is injected into the groundwater saturated matrix. PXY-1006, Abstract, Claim 6; PXY-1011, ¶ 56.

As with zero valent metals, the use of organic hydrogen donors was both known and practiced in the United States prior to January 2003. For example, a field test was conducted in August of 2000 at Offutt Air Force base in Omaha, Nebraska, which favorably determined the efficacy of organic mulch for promoting biological deductive dechlorination of ground water. PXY-1005, p. 1; PXY-1011, ¶ 57.

### **3.** The use of zero valent metals in combination with organic hydrogen donors was known in the art

Before the priority date, the symbiotic relationship between the use of a zero valent metal and the use of an organic hydrogen donor was also understood. PXY-1011, ¶ 58. Several prior art references reflect using both a zero valent metal and an organic hydrogen donor in soil remediation. As will be described in further

detail in Section VII below, Vance describes a dechlorination method of injecting zero valent iron along with a carbohydrate (organic hydrogen donor), specifically corn syrup, into the soil. Hitchens, Alvarez, Igawa, Cantrell and Day all describe introducing zero valent metals and organic hydrogen donors for dehalogenation/dechlorination methods for soil remediation. PXY-1011, ¶¶ 58-61.

Methods of dechlorination using both zero valent metals and organic hydrogen donors were also practiced across the country prior to January 2003. For example, as early as 1995, the W.R. Grace site in Charleston, South Carolina, was treated with organic amendments and iron powder for the same purpose described in the '709 Patent. PXY-1017, p. 12; PXY-1011, ¶ 63.

Similarly, organic amendments and iron powder were introduced to contaminated soil at the Iowa Army Ammunition Plant in Burlington, Iowa, in August 2001, and at the Joliet Army Ammunition Plant, the Yorktown Naval Weapons Station, and the Hawthorne Army Depot prior to July 2002 for the same purpose described in the '709 Patent. PXY-1005, p. 3; PXY-1011, ¶ 63.

Injection and fracturing processes were also well known. For example, TNT describes injecting zero valent iron powder and water through an injection rod in the ground into the treatment zone with pressure and Day describes fracturing to create channels for iron slurry. PXY-1005, p. 4, Figure 3; PXY-1012, pp. 6-7 PXY-1011, ¶ 44-46.

#### VII. THE CHALLENGED CLAIMS ARE NOT PATENTABLE

#### A. Statutory Grounds for Challenges

Claims 1-16 of the '709 Patent are anticipated under 35 U.S.C. § 102(a), (b) and/or (e) (pre- AIA) and/or obvious under 35 U.S.C. § 103(a) (pre- AIA) in view of the prior art listed in the following table:

Ground	35 U.S.C.	Index of Reference(s)	<b>'263</b> Patent Claims
1	§ 102(b)	Hitchens	1, 7, 9 and 14
2	§ 102(e)	Vance et al.	1, 3, 7, 9, and 14-16
3	§ 102(b)	Alvarez et al.	1, 3, 7 and 14
4	§ 103(a)	Rice in view of Cantrell	1-3, 5, 7, 11, 12 and 14-16
5	§ 103(a)	Vance in view of Schuring	4, 10 and 13
6	§ 103(a)	Hitchens in view of Grasso	8
7	§ 103(a)	Hitchens in view of Odom	6

Sections VII.B through VII.I below identify where each element of each Challenged Claim is found in the prior art references in compliance with 37 C.F.R. § 42.104(b)(4). Exhibits PXY-1001 through PXY-1026 are attached to provide the supporting evidence relied upon to support the challenges in compliance with 37 C.F.R. § 42.104(b)(5). In further support of this Petition, Petitioner includes a Declaration of its technical expert Dr. John A. Bergendahl, attached as Exhibit PXY-1011. Taken together, this evidence establishes a reasonable likelihood that Petitioner will prevail as to the Challenged Claims.

#### B. Statement of Non-Redundancy of the Grounds.

This Petition sets forth seven Grounds challenging the patentability of the Challenged Claims. Petitioner submits that none of the Grounds are redundant as each Ground includes a separate statutory basis and/or is directed to a different set of the sixteen Challenged Claims. *Sure-Fire Elec. Corp. v. Shenzen El Ltg. Co. Ltd.*, IPR2014-01448, Paper 25, 4-5 (June 25, 2015).

As shown in the chart below, there are four separate grounds of rejection directed to independent Claim 1, all of which are necessitated by separate statutory challenges to Claim 3 or to support singular challenges to Claims 4-6, 8, and 10-13, respectively. For example, the challenges to Claim 8 (Ground 6) and Claim 6 (Ground 7) are predicated on the challenge to Claims 1 and 7 in Ground 1; the challenge to Claims 4, 10 and 13 in Ground 6 is predicated on the challenge to Claims 1 and 3 in Ground 2; and the challenge to Claims 2, 5,11 and 12 of Ground 4 is predicated on the challenge to Claims 1 and 3 in Ground 4.

There are also three separate challenges to Claim 3, all of which are based on separate statutory grounds and/or are necessary to support the singular challenges to Claims 4 and 10-13. The challenge to Claim 3 under Ground 2 relies upon a § 102(a) reference and is subject to swear behind, whereas the challenge to Claim 3 under Challenge 3 relies upon a § 102(b) reference. No claim is twice challenged under the same statutory basis with the exception of Claim 1 as discussed above, and the trivial limitations of Claims 7 and 14. Claims 7 and 14 add no measurable burden upon the Board or the Patent Owner as any patentable basis for these claims are overcome by the challenges to Claim 1 in Grounds 1-4, respectively.



#### C. Ground 1: Claims 1, 7, 9 and 14 are anticipated under 35 U.S.C. § 102(b) by Hitchens

#### 1. Overview of Hitchens

U.S. Patent No. 6,265,205 to Hitchens *et al.*, titled "ENHANCEMENT OF SOIL AND GROUNDWATER REMEDIATION," filed on January 27, 1998 and issued on July 24, 2001, qualifies as prior art under 35 U.S.C. § 102(b).

Hitchens discloses a method for enhancing the biodegradation of chlorinated organic compounds in the ground by naturally occurring bacteria using a zero valent metal and organic hydrogen donor. PXY-1003, 1:10-15; PXY-1011, ¶ 77. Claims 42-44 of Hitchens provide a tracing of a method corresponding to the method recited in the Challenged Claims:

42. A method for enhancing the rate of biodegradation of chlorinated organic compounds in a contaminated environment comprising: generating hydrogen in situ [wherein the step of generating hydrogen includes corroding metal particles dispensed in the soil (Claim 43)] dissolving at least one organic electron donor in water, wherein the at least one organic electron donor is selected from glucose... delivering the water into the environment by electroosmosis and ...[delivering nutrients into the soil. (Claim 44)]. PXY-1003,17:8-18:13.

Hitchens discloses a method for soil and ground water remediation utilizing both *biotic* and *abiotic* mechanisms. PXY-1011, ¶ 78. Regarding the biotic

mechanism, Hitchens discloses the advantages of "supplying hydrogen (H<sub>2</sub>) to naturally occurring anaerobic bacteria in the soil or ground water. These organisms use the hydrogen as an electron donor to perform adventitious chemical reactions including the dehalogenation of chlorinated organic compounds." PXY-1003 Abstract; PXY-1011, ¶ 79. Hitchens also describes that chlorinated solvents can be degraded in abiotic reactions with granular iron. The reactions include:  $Fe^{\circ} \rightarrow Fe^{2+}+2e^{-}$ ; C<sub>2</sub>HCl<sub>3</sub>+3H<sup>+</sup>+6 $e^{-} \rightarrow C_2H_4$ +3Cl<sup>-</sup> and the subsequent formation of hydrogen gas 2H<sub>2</sub>O+2 $e^{-} \rightarrow H_2(g)$ +2OH<sup>-</sup>. PXY-1003, 3:39-56; PXY-1011, ¶ 80.

Hitchens recognizes the symbiotic relationship between the presence of a zero valent metal and an organic hydrogen donor, "this chemical degradation process [abiotic] is assisted by a parallel biological [biotic] route where the growth of methanogenic bacteria is stimulated by the produced hydrogen, and these organisms enzymatically dechlorinating additional contaminants" and identifies that the availability of a suitable electron donor (organic hydrogen donor) for this process is critical. PXY-1003, 3:57-60; PXY-1011, ¶ 81.

Under anaerobic conditions, all chlorinated ethenes can be completely transformed into benign end products through sequential reductive dechlorination. In this biologically catalyzed process, chlorinated pollutants act as electron acceptors, the chloride moiety is removed from molecules and replaced by hydrogen. The availability of a suitable FIG. 1



24

DM2\6357536.1

electron donor for this process is one of the key rate limiting elements. PXY-1003, 2:1-8.

Fig 1. Illustrates this mechanism:

Hitchens also discloses "vitamin  $B_{12}$ (co-containing porphyrin) which is often found in bacteria, and coenzyme  $F_{430}$  (Ni-containing porphyrin), found only in methanogenic bacteria have the capacity to mediate the eight electron sequential reduction of PCE to ETH" as shown above. PXY-1003, 2:63-67; PXY-1011, ¶ 83.

Hitchens describes that organic hydrogen donors, like glucose, lactate and formates are advantageous in supporting reductive transformations of perchloroethylene (PCE). PXY-1003, 2:32-44; PXY-1011, ¶ 84.

Sustaining reductive dechlorination is highly dependent upon the availability of an electron donor (reductant) in the contaminated environment. Some of the compounds that have been found to support the reductive transformation of PCE are glucose, acetate, formate, methanol, lactate, propionate, crotonate, butyrate, ethanol, and other compounds such as toluene and dichloromethane. Higher dechlorinating activities were observed when organic substrates that contain more reducing power during anaerobic digestion (e.g. formate, glucose, lactate) were used. Also, electron donors that produce hydrogen more slowly give a selective advantage to organisms that dechlorinate chlorocarbons over those that generate methane. PXY-1003, 2:32-44.

25

## Detailed Analysis of the Challenged Claims in View of Hitchens a. Claim 1

#### *i.* A method for accelerated anaerobic dechlorination of subsoil matter, comprising the steps of:

To the extent the preamble is limiting, Hitchens describes the toxicity of many contaminants can be decreased or eliminated by reductive dechlorination, which involves successive shedding of chlorine atoms under reduced anaerobic condition that is biologically catalyzed. PXY-1003, 1:18-25; PXY-1011, ¶ 85 [1.0]. Hitchens discloses "This invention relates to the field of bioremediation of contaminated environments. The invention particularly relates to a method for enhancing the biodegradation of chlorinated organic compounds in the ground by naturally occurring bacteria." PXY-1003, 1:10-15; PXY-1011, ¶ 85 [1.0]. Hitchens discloses a method for accelerated anaerobic dechlorination of subsoil matter. PXY-1011, ¶ 85 [1.0].

#### *ii.* supplying a mixture including a zero valent metal into soil pathways to biologically react with the dissolved chlorinated solvents in the groundwater; and

Hitchens discloses "[c]hlorinated solvents, such as trichloroethylene and perchloroethylene can be degraded by reactions with **granular iron**. This abiotic reductive dehalogenation is thought to proceed with pseudo-first order kinetics" PXY-1003, 3:39-56; PXY-1011, ¶ 85 [1.1]. Hitchens discloses the governing

equations for the reductive dehalogenation (dechlorination) includes a mixture of zero valent iron and magnesium. PXY-1011, ¶ 85 [1.1].

Fe<sup>o</sup>
$$\rightarrow$$
Fe<sup>2+</sup>+2e<sup>-</sup> (1)  
C<sub>2</sub>HCl<sub>3</sub>+3H<sup>+</sup>+6e<sup>-</sup> $\rightarrow$ C<sub>2</sub>H<sub>4</sub>+3Cl<sup>-</sup> (2) PXY-1003.3:49-51

Hitchens further discloses "TCE  $[C_2HCl_3]$  degrades spontaneously in the presence of both water and iron, requiring no additives or application of energy." PXY-1003, 4:1-3; PXY-1011, ¶ 85 [1.1]. In Hitchens, the "iron is placed deep enough to intercept the saturated thickness of the plume in a contaminated zone." PXY-1003, 4:17-19. Fig. 7 illustrates injection wells in which the zero valent iron is placed in contact with the ground water. PXY-1003, Fig 7; PXY-1011, ¶ 85

[1.1]. Hitchens also describes how particles used in the invention may be distributed in the soil by placing them in a trench, injecting them into holes or wells, or packaging them into porous filter socks. PXY-1003,12:18-29; PXY-1011, ¶



85 [1.1].

The zero valent iron in the mixture is involved in a multitude of reactions, including reactions with the dissolved chlorinated solvents in the groundwater.

PXY-1011, ¶ 85 [1.1]. The addition of zero valent iron to the subsurface that is under reducing conditions (anaerobic) will result in the formation of H<sub>2</sub> gas as disclosed by Hitchens. PXY-1003, 11:533-41; PXY-1011, ¶ 85 [1.1]. This hydrogen gas produced enhances the anaerobic biodegradation process of the dissolved chlorinated solvents in the soil by indigenous anaerobic bacteria. PXY-1003, 1:10-15; PXY-1011, ¶ 85 [1.1].

> *iii.* supplying an organic hydrogen donor into the soil pathways to produce dechlorinating conditions such that indigenous anaerobic bacteria biodegrade residual concentrations of chlorinated solvents.

Hitchens discloses "supplying hydrogen (H<sub>2</sub>) to naturally occurring anaerobic bacteria in the soil or ground water. These organisms use the hydrogen as an electron donor to perform adventitious chemical reactions including the dehalogenation of chlorinated organic compounds." PXY-1003, Abstract; PXY-1011, ¶ 85 [1.2]. Hitchens discloses the organic hydrogen donors [electron donors] that advantageously support dechlorination "are glucose, acetate, formate, methanol, lactate, propionate, crotonate, butyrate, ethanol, and other compounds such as toluene and dichloromethane. Higher dechlorinating activities were observed when organic substrates that contain more reducing power during anaerobic digestion (e.g. formate, glucose, and lactate) were used." PXY-1003, 2:32-44; PXY-1011, ¶ 85 [1.2]. As described by Hitchens, supplying an organic hydrogen donor, leads and is capable of leading to the production of hydrogen and the dechlorinating conditions that allow indigenous anaerobic bacteria to biologically degrade residual chlorinated solvents. PXY-1011, ¶ 85 [1.2].

#### **b.** Claim 14 the method of Claim 1 wherein said metal is iron.

As discussed above, "[c]hlorinated solvents, such as trichloroethylene and perchloroethylene can be degraded by reactions with **granular iron**." PXY-1003, 3:39-56; PXY-1011, ¶ 85 [14.0].

Fe<sup>o</sup> $\rightarrow$ Fe<sup>2+</sup>+2e<sup>-</sup> (1) C<sub>2</sub>HCl<sub>3</sub>+3H<sup>+</sup>+6e<sup>-</sup> $\rightarrow$ C<sub>2</sub>H<sub>4</sub>+3Cl<sup>-</sup> (2) PXY-1003,3:49-51

Hitchens discloses the zero valent metal used for reductive dechlorination is iron (Fe°). PXY-1011, ¶ 85 [14.0].

### <u>c. Claim 7</u> the method of claim 1 wherein the mixture further includes nutrients.

Hitchens discloses the step of "delivering nutrients into the soil" (PXY-

1003, 16:33-34) in addition to the elemental iron. PXY-1011, ¶ 85 [7.0].

d. Claim 9	The method of claim 1 wherein said organic
	hydrogen donor is from the group consisting of
	lactate, propionate, chitin, butyrate, acetate,
	sugars, glycerol tripolylactate,
	xylitol pentapolylactate, and sorbitol
	hexapolylactate.

Hitchens discloses the organic hydrogen donors that advantageously support dechlorination "are glucose, *acetate*, formate, methanol, *lactate, propionate*, crotonate, *butyrate*, ethanol, and other compounds such as toluene and dichloromethane. Higher dechlorinating activities were observed when organic substrates that contain more reducing power during anaerobic digestion (e.g. formate, glucose, and *lactate*) were used." PXY-1003, 2:32-44 (emphasis added); PXY-1011, ¶ 85 [9.0].

# D. Ground 2: Claims 1, 3, 7, 9 and 14-16 are anticipated under 35 U.S.C. § 102(e) by U.S. Patent Application Publication No. 2002/0151602

**1.** Overview of Vance et al.

U.S. Patent Application Publication No. 2002/0151602 to Vance et al., titled "METHOD OF MAKING AND USING NANOSCALE METAL" filed on December 19, 2001, claiming priority to Provisional Application No. 60/257,917 filed December 19, 2000, and published October 17, 2002 qualifies as prior art under 35 U.S.C. §§ 102(a) and (e).

Vance discloses a method for soil and ground water remediation utilizing a naturally occurring reaction that takes place between elemental iron and chlorinated compounds, where the chlorinated compounds are reduced to harmless compounds. PXY-1010, Abstract; PXY-1011,¶ 86. Specifically, Vance describes injecting under pressure a colloidal suspension made up of elemental iron and corn

syrup into the soil. PXY-1010, Claims 11-20; PXY-1011, ¶ 86. Corn syrup contains sugars, and the '709 Patent expressly identifies "sugars" as an organic hydrogen donor. PXY-1001, 4:29-32,6:57-60; PXY-1011, ¶ 86.

#### 2. Detailed Analysis of the Challenged Claims in View of Vance

#### a. Claim 1

#### *i.* A method for accelerated anaerobic dechlorination of subsoil matter, comprising the steps of:

To the extent the preamble is limiting, Vance is directed to a method of in situ treatment of chlorinated solvents "based on the sequential reduction of chlorinated hydrocarbons to innocuous end products such as methane, ethane or ethene. PXY-1010, ¶ [0002]; PXY-1011, ¶ 94 [1.0]. The process utilized by Vance "may be best described as anaerobic corrosion of the metal by the chlorinated hydrocarbon." PXY-1010, ¶ [0003]; PXY-1011, ¶ 94 [1.0].

#### *ii.* supplying a mixture including a zero valent metal into soil pathways to biologically react with the dissolved chlorinated solvents in the groundwater; and
Vance describes injecting/supplying a colloid suspension having elemental metal<sup>2</sup> "mixed with a second metal", a carbohydrate, nitrogen and/or sand, into the contaminated soil for "the sequential reduction of chlorinated hydrocarbons to innocuous end products such as methane, ethane or ethene." PXY-1010, ¶ [0002], [0113], [0118]-[0121], Claims 11, 12, 13 and 18; PXY-1011, ¶ 94 [1.1].

Additionally, the present invention is to a method of injecting nanoscale metal particles into soil, comprising the steps of: a. making a colloid suspension having metal particles in the presence of a carbohydrate; and b. injecting said colloid suspension into the soil through a well at a flow rate sufficient to move the colloid suspension through the soil. PXY-1010, ¶¶ [0118]-[0120].

The nanoscale metal particles may be injected by any known method but are preferably injected under pressure (without the use of surfactants) or injected with hydraulic pressure. Most preferably, nitrogen under pressure will be used. The elemental metal or bimetallic colloid may [be] used alone or may be suspended in the nitrogen gas and may be mixed with a carbohydrate solution (also called carrier). PXY-1010, ¶ [0121].

32

<sup>&</sup>lt;sup>2</sup> Elemental metal is any metal in its elemental state having a zero valence. Metals that are preferred are selected from the group of iron, tin, zinc and palladium. The most preferred is iron. PXY-1010,  $\P$  [0112].

Vance in Fig. 5 (truncated) provides an example of supplying a mixture including elemental iron into the soil. PXY-1010, Fig. 5; PXY-1011, ¶ 94 [1.1].

The zero valent iron in the mixture is involved in a multitude of reactions, including reactions with the dissolved chlorinated solvents in the groundwater. PXY-1011, ¶ 94 [1.1]. The addition of zero valent iron to the



**FIG.5** (Truncated)

subsurface that is under reducing conditions (anaerobic conditions as described by Vance) will result in the formation of H<sub>2</sub> gas. PXY-1011, ¶ 94 [1.1]. The hydrogen gas enhances the anaerobic biodegradation process of the dissolved chlorinated solvents in the soil by indigenous anaerobic bacteria as is well known in the art. PXY-1011, ¶ 94 [1.1].

*iii.* supplying an organic hydrogen donor into the soil pathways to produce dechlorinating conditions such that indigenous anaerobic bacteria biodegrade residual concentrations of chlorinated solvents.

Vance describes supplying a colloidal suspension of iron and an organic hydrogen donor, specifically corn syrup, into the soil pathways. PXY-1010, ¶¶ [0118]-[0122], Fig. 5, Claims 11, 15 and 18; PXY-1011, ¶ 94 [1.2].

Additionally, the present invention is to a method of injecting nanoscale metal particles into soil, comprising the steps of: a. making a colloid suspension having metal particles in the presence of a carbohydrate; and b. injecting said colloid suspension into the soil through a well at a flow rate sufficient to move the colloid suspension through the soil. PXY-1010,  $\P$  [0118]-[0120].

Any carbohydrate solution that creates an oxygen scavenging environment may be mixed with the metal. Most preferably, corn syrup may be used in suspension with nanoscale metal and injected by pressure into a contaminated sub-soil zone. The corn syrup helps to prevent elemental iron from rusting which would have the effect of lessening the remediation capability of the iron. PXY-1010, ¶ [0122].

As known by a POSA, an oxygen scavenging environment (anaerobic environment) which is naturally produced from the introduction of the organic hydrogen donor (corn syrup) is an environment in which indigenous anaerobic bacteria may and do biologically degrade residual concentrations of chlorinated solvents. PXY-1011, ¶ 94 [1.2].

#### **b.** Claim 14 the method of claim 1 wherein said metal is iron.

Vance discloses the metal is iron in meeting the limitations of Claim 1. *See VII.C.2.a.ii* (above). Vance claims the use of iron as the elemental metal (zero valent metal). PXY-1010, Claim 2; PXY-1011, ¶ 94 [14.0].

2. The method of claim 1, wherein said elemental metal is selected from the group consisting of iron, tin, zinc and palladium and mixtures thereof. PXY-1010, Claim 2.

wherein the steps of supplying said mixture and said organic hydrogen donor are carried out by

**c.** 

Claim 3

placing an injection rod into the soil and then injecting them under pressure through an injection rod.

Vance describes an injection system with an injection rod 92 (annotated in red), pump 90 (annotated in yellow) in which to inject the elemental iron and corn syrup under pressure into the bore hole 82 (annotated in blue) in the soil 94 (annotated in brown). PXY-1010, ¶ [0102], [0104], [0121], Figs. 2, 4-5, Claim 16; PXY-1011, ¶ 94 [3.0].

The injection may also be achieved with the use of hydraulic pressure in which a slurry of elemental metal or bimetallic colloid and carbohydrate solution are injected. PXY-1010,  $\P$  [0121].



FIG. 4

PXY-1010 Fig. 4 (annotated)

FIG. 4 shows another embodiment of a method of the present invention 80 for injecting the nanoscale metal 96 of the present invention by pump 90 through an injector 92 into a[n] open bore hole 82 through a screen 86 into soil beneath ground level 88 and beneath the water table 84 with the use of hydraulic pressure 98 for fracturing the soil 94. The hydraulic pressure 98 is used to fracture the soil 94 and the nanoscale metal 96 is injected either simultaneously or after the hydraulic pressure 98 has been applied. PXY-1010, ¶ [0104]

Vance discloses all the limitations of Claim 3. PXY-1011, ¶ 94 [3.0].

### d.Claim 7the method of claim 1 wherein the mixture<br/>further includes nutrients.

Vance further describes along with the zero valent iron supplying in the colloidal, a carbohydrate atomized in nitrogen. PXY-1010, claim 20; PXY-1011, ¶ 94 [7.0].

The method of claim 11, wherein said carbohydrate is added in atomized form by injecting said carbohydrate into compressed nitrogen gas. PXY-1010, Claim 20.

As described in the '709 Patent, nitrogen is a nutrient. PXY-1001, 2:64-67;

### PXY-1011, ¶ 94 [7.0].

A solution of sodium sulfite and nutrients (nitrogen and orthophosphate) is immediately injected into the subsurface pathways and voids that were developed during the gas injection step. PXY-1001, 2:64-67.

<u>e. Claim 9</u> The method of claim 1 wherein said organic hydrogen donor is from the group consisting of lactate, propionate, chitin, butyrate, acetate, sugars, glycerol tripolylactate, xylitol pentapolylactate, and sorbitol hexapolylactate.

Corn syrup is a known *sugar*. PXY-1011, ¶ 94 [9.0].

# f.Claims 15 and 16The method of claim 1 wherein said metal is in a<br/>colloidal suspension; and The method of claim<br/>15 wherein the colloidal suspension includes a<br/>reducing agent.

Vance as described above (See VII.C.2.a.ii and iii) discloses a colloidal

suspension of iron powder and corn syrup. PXY-1010, ¶¶ [0118]-[0120], Claim

18; PXY-1011, ¶ 94 [15.0]. Corn syrup is a known reducing agent. PXY-1011, ¶

94 [16.0].

- E. Ground 3: Claims 1, 3, 7 and 14 are anticipated under 35 U.S.C. § 102(b) by Alvarez et al.
- 1. Overview of Alvarez

PCT Published Application WO 98/49106 to Alvarez et al., titled "FE(0)

BASED BIOREMEDIATION OF AQUIFERS CONTAMINATED WITH MIXED WASTES" was filed April 25, 1998 with a priority date of April 25, 1997. Alvarez Published on November 5, 1998 and qualifies as prior art under at least 35 U.S.C. § 102 (b). Alvarez discloses a method "for the bioremediation of environmental sites and aqueous solutions using a synergistic combination of biotic and abiotic processes." PXY-1004, p. 9; PXY-1011, ¶ 95. The method being a "reductive treatment using zero valent iron and anaerobic biotransformations." PXY-1004, Abstract; PXY-1011 ¶ 95. Alvarez describes well systems, as illustrated below in Fig. 2, "involve injection of fluids or fluid/particulate mixtures for distribution into a treatment zone within the target area of the aquifer." PXY-1004, p. 16; PXY-1011, ¶ 97.

Alvarez describes in situ remediation may be achieved using a combination/mixture which includes iron and is placed in the path of a contaminant plume, either in a trench buried as a broad continuous curtain, or injected as colloids. PXY-1004, p. 17; PXY-1011, ¶ 98. The particles, or combination, injected by Alvarez are zero





valent metals "preferably iron, iron alloy, or iron bimetal" in the form of powder, beads, pellets, etc. PXY-1004, p. 10; PXY-1011, ¶ 99. In addition "the composition may be augmented to provide one or more nutrients, vitamins, minerals, or substrates for utilization by the microbial colony." PXY-1004, p. 11. The substrate being an electron donor such as methanol or acetate. PXY-1004, p. 23-24; PXY-1011, ¶ 99. Alvarez discloses injecting zero valent iron and an organic hydrogen donor into the soil pathways for soil dehalogenation as described in the Challenged Claims. PXY-1011, ¶ 100.

#### 2. Detailed Analysis of the Challenged Claims in View of Alvarez

#### a. Claim 1

### *i.* A method for accelerated anaerobic dechlorination of subsoil matter, comprising the steps of:

To the extent the preamble is limiting, Alvarez discloses "method, devices and apparatus for bioremediation of mixed waste water aquifers" and "methods for in situ and ex situ remediation of groundwater and waste water." PXY-1004, abstract; PXY-1011, ¶ 101 [1.0]. Alvarez discloses the method results in reductive dechlorination, stating "[p]olychlorinated organics can also be reduced using the [electron] generated by iron corrosion by replacing Cl atoms with hydrogen atoms. This form of reductive dechlorination is termed hydrogenolysis (Vogel et al., 1987)." PXY-1004, p. 4; PXY-1011, ¶ 101 [1.0]. The dechlorination method is anaerobic and Alvarez notes "[a]naerobic conditions are required to produce the H<sub>2</sub> from F(0) corrosion and support the growth of useful anaerobic bacteria." PXY-1004, p. 6; PXY-1011, ¶ 101 [1.0].

### *ii.* supplying a mixture including a zero valent metal into soil pathways to biologically react with the dissolved chlorinated solvents in the groundwater; and

Alvarez's dechlorination method supplies a composition/mixture containing zero-valent iron and bacteria culture to make contact with a contaminant plume in the ground. PXY-1004, pp. 6-11, 17, Claim 35; PXY-1011, ¶ 101 [1.1].

in situ remediation may be achieved using barrier technologies such as that of and the compositions of the present invention may also be placed in the path of a contaminant plume, either on a trench (O'Hannesin and Gillham, 1992), buried as a broad continuous curtain (Blowes et al., 1995), or injected as colloids (Kaplan et al., 1994). PXY-1004, p. 17.

The zero-valent iron of the composition is preferably iron, iron alloy, or iron bimetal. in [sic] It may be in the form of metal filings, shavings, turnings, powder, mesh, steel wool, beads, rods, or pellets (exemplary sources include e.g., Malinkrodt, Fisher Scientific, Master Builder, Aldrich Chemical Co., scrap metal suppliers, and the like). Iron bimetallics, composed of a combination of Fe(O) and any of the following: Ni(O), Zn(O), Pt(O), and Pd(O), are also desirable for the compositions and processes of the invention, as are iron-based metallic alloys. PXY-1004, p. 10.

[T]etrachloroethene (PCE) and TCE can be used as electron acceptors for growth of some anaerobic organisms with the end products being ethene or ethane (Bolliger et al., 1993; Bolliger, 1995; Scholz-Muramatsu et al., 1995). Anaerobic conditions are required to produce the H2 from Fe<sup>(O)</sup> corrosion and support the growth of useful anaerobic bacteria. PXY-1004, p. 7-8. [T]he invention concerns iron-supported autotrophic methods which utilize a device comprising a composition containing zero-valent iron and a culture of one or more species of hydrogenotrophic bacteria to remove target contaminants. PXY-1004, p. 9.

The zero valent iron in the composition/mixture are involved with many reactions in the subsurface, including reactions with the dissolved chlorinated solvents in the groundwater. PXY-1011, ¶ 101 [1.1]. The addition of zero valent iron to the subsurface that is under reducing conditions (anaerobic) will result in the formation of H<sub>2</sub> gas as disclosed by Alvarez. PXY-1004, p. 6; PXY-1011, ¶ 101 [1.1]. This hydrogen gas enhances the anaerobic biological degradation process of the residual dissolved chlorinated solvents by the indigenous anaerobic bacteria. PXY-1011, ¶ 101 [1.1]. The dechlorination method is anaerobic and the indigenous anaerobic bacteria in the soil biologically degrade the dissolved chlorinated solvents. *Id.*, ¶ 101 [1.1]. The mixture/composition as described in Alvarez also contains a culture of hydrogenotrophic bacteria to remove target contaminants (chlorinated solvents) in the soil. *Id.*, ¶ 101 [1.1].

### *iii.* supplying an organic hydrogen donor into the soil pathways to produce dechlorinating conditions such that indigenous anaerobic bacteria biodegrade residual concentrations of chlorinated solvents.

Alvarez discloses "the composition may be augmented to provide one or more nutrients, vitamins, minerals, or substrates for utilization by the microbial colony." PXY-1004, p. 11. Alvarez notes "organic substrates that are commonly used to support biological denitrification" are methanol and acetate. PXY-1004, p. 26; PXY-1011, ¶ 101 [1.2]. In addition, because reductive dechlorination is an electron consuming process, Alvarez describes, "the stimulation of which by the addition of an exogenous electron donor is common (Bagley and Gossett, 1995; 15 Freedman and Gossett, 1989; Fathepure and Boyd, 1988)." PXY-1004, p. 58; PXY-1011, ¶ 101 [1.2]. Methanol and acetate are described as electron donors by Alvarez. PXY-1004, pp. 23-24; PXY-1011, ¶ 101 [1.2].

Electron donors that have been investigated for methanogenic biotransformation of CAHs include *methanol* (Mikesell and Boyd, 1990; Bagley and Gossett, 1995), *acetate* (Bouwer et al., 1981; Bouwer and McCarty, 1983; Hughes 10 and Parkin, 1996), and hydrogen (DiStefano et al., 1992). (Emphasis added) PXY-1004, pp. 23-24.

As described by Alvarez supplying acetate (an organic hydrogen donor) stimulates the dechlorination process by providing electrons that allow indigenous anaerobic bacteria to biologically degrade residual chlorinated solvents. PXY-1011, ¶ 101 [1.2].

#### **b.** Claim 14 The method of claim 1 wherein said metal is iron.

Alvarez discloses the zero valent metal is iron, "The zero-valent iron of the composition is preferably iron, iron alloy, or iron bimetal. "PXY-1004, p. 10; PXY-1011, ¶ 101 [14.0].

### <u>c. Claim 3</u> the method of Claim 1 wherein the steps of supplying said mixture and said organic hydrogen donor are carried out by placing an injection rod into the soil and then injecting them under pressure through an injection rod.

Alvarez describes the injection of the composition into the ground using an injection system.

[T]he invention provides methods and apparatus using an injected treatment zone. A treatment zone which (unlike the treatment wall) is not confined within strict boundaries can be established by using injection wells or by hydraulic fracturing (FIG. 3). Well systems typically involve injection of fluids or fluid/particulate mixtures for distribution into a treatment zone within the target area of the aquifer (Vidic and Pohland, 1996). PXY-1004, p. 16.

It is inherent that the injection process utilizing injection wells or hydraulic fracturing as described in Alvarez and known in the art, necessarily utilizes an injection rod, pipe, or tube, etc. to introduce fluids, gas or particulate mixtures for distribution into the treatment zone and would necessarily inject the fluids, gas or particulate mixtures into the ground under pressure. PXY-1011, ¶ 101 [3.0].

### d.Claim 7the method of claim 1 wherein the mixture<br/>further includes nutrients.

Alvarez describes the addition of nutrients in the composition/mixture with

zero valent metal. PXY-1004, p. 11; PXY-1011, ¶ 101 [7.0].

Also optionally, the composition may be augmented to provide one or more nutrients, vitamins, minerals, or substrates for utilization by the microbial colony. PXY-1004, p. 11.

### F. Ground 4: Claims 1-3, 5, 7, 11, 12 and 14-16 are rendered obvious under 35 U.S.C. § 103 by Rice in view of Cantrell

#### **1.** The Rice reference

U.S. Patent No. 6,001,252 to Rice et al., titled "IN SITU ANAEROBIC

DEHALOGENATION" filed on July 9, 1998 and issued on December 14, 1999,

qualifies as prior art under 35 U.S.C. § 102(b).

Rice describes "a method for in situ anaerobic dehalogenation of a halogenated organic compound in a ground water plume" in which a deoxygenated aqueous solution including an organic hydrogen donor (lactate, acetate or formate) is injected into the groundwater saturated matrix. PXY-1006, Abstract, Claim 6; PXY-1011, ¶ 103.

#### 2. The Cantrell reference

U.S. Patent No. 5,857,810 to Cantrell et al., titled "IN-SITU CHEMICAL BARRIER AND METHOD OF MAKING" filed on February 27, 1997 and issued on January 12, 1999, qualifies as prior art under 35 U.S.C. § 102(b) ("Cantrell") (attached herein as Exhibit PXY-1026).

Cantrell describes the treatment of contaminated soil with the injection of iron (Fe<sup>(0)</sup>) as a colloidal suspension into the subsurface to remediate contaminants, such as halogenated hydrocarbon compounds. PXY-1026, 2:11-19, 5:17-35; PXY-1011, ¶ 109.

### 3. Detailed Analysis of the Challenged Claims in View of Prior Art References

#### <u>a. Claim 1</u>

### *i.* A method for accelerated anaerobic dechlorination of subsoil matter, comprising the steps of:

Rice discloses in situ anaerobic dechlorination of a ground water plume.

PXY-1006, Abstract; PXY-1011, ¶ 114 [1.0].

A method for in situ anaerobic dehalogenation of a halogenated organic compound in a groundwater plume reduces or prevents indigenous aerobic microorganisms from competing for a supplied electron donor with an anaerobic microorganism that reductively dehalogenates the organic compound when an electron donor is available. PXY-1006, Abstract.

*ii.* supplying a mixture including a zero valent metal into soil pathways to biologically react with the dissolved chlorinated solvents in the groundwater; and Rice discloses supplying a mixture to biologically react with the dissolved solvents in the ground water, including a substrate and if necessary suitable anaerobic microorganisms; the mixture, however, does not include zero valent metal. PXY-1006, 4:9-10, 29-43; PXY-1011, ¶ 114 [1.1].

Cantrell discloses a method to dechlorinate dissolved chlorinated solvents in ground water which includes the introduction of zero valent metal into the soil pathways. PXY-1026, 2:11-19; 5:17-35, Claims 1, 13; PXY-1011, ¶ 114 [1.1].

Specifically, Cantrell describes "[w]hen zero-valent iron metal particles are used as the solid particles, suspensions of Fe<sup>0</sup> colloids  $(2\pm 1 \ \mu\text{m})$  are injected at a colloid injection rate, P $\leq$ 0.01, and concentration, P~0.05, to uniformly distribute colloids throughout soil columns at concentrations >0.3% (w/w)." PXY-1026, 5:55-60; PXY-1011, ¶ 114 [1.1]. Cantrell states the selection of materials is dependent on the targeted contaminants and that "[z]ero-valent iron is preferred for its ability to remediate a wide range of groundwater contaminants. It is an extremely strong chemical reductant and has the capability to dehalogenate several halogenated-hydrocarbon compounds." PXY-1026, 5:17-35; PXY-1011 ¶ 114 [1.1].

Cantrell further describes many experiments utilizing zero valent colloids: An experiment was conducted to evaluate the ability of a commercially available Fe<sup>0</sup> colloid (Product Number S3700; International Specialty Products, Wayne, N.J.) to remove an oxidized dissolved species from solution." PXY-1026, 6:50-54.

Suspensions of  $Fe^0$  colloids were introduced into sand columns at several flow rates and influent colloid concentration. *Id.*, 8 59-61.

An experiment was conducted to demonstrate three shearthinning fluids with  $Fe^0$  as colloids for emplacement in columns containing coarse-grained sand. *Id.*, 10:57-59.

To test the effectiveness of the VP for enhancing the emplacement of  $Fe^{0}$  colloids in porous media for greater distances, a 3-m long column experiment was conducted. In this case, the suspension of 1.0%  $Fe^{0}$  colloids in 0.01% VP was injected at a darcy velocity of 0.154 cm sec-1 and a three pore volume throughput (FIG. 7). *Id.*, 13:62-67.

*iii.* supplying an organic hydrogen donor into the soil pathways to produce dechlorinating conditions such that indigenous anaerobic bacteria biodegrade residual concentrations of chlorinated solvents.

Rice discloses supplying organic hydrogen donors, lactate, propionate and acetate in to the soil. PXY-1006, Abstract, 4:9-10, Claims 1, 6; PXY-1011, ¶ 114 [1.2].

The method includes the step of injecting in situ into a groundwater saturated matrix ... a deoxygenated aqueous solution that comprises an electron donor to facilitate reductive dehalogenation of the organic compound. PXY-1006, Abstract, 4: 9-10.

The electron donor substrate should be a relatively simple organic molecule having available hydrogens that can act as the proximate electron donor during dehalogenation. Preferred electron donors include pyruvate, lactate, formate, propionate and acetate. PXY-1006, 4: 9-10.

Given the teachings of Rice and Cantrell, it would have been obvious to a POSA to include a zero valent iron in a colloidal suspension with Rice's aqueous solution. PXY-1011, ¶ 111. A POSA would have multiple reasons to combine the teachings of Rice and Cantrell: 1) the addition of zero valent iron to Rice's solution provides an additional (abiotic) mechanism to reductively dechlorinate chlorinated solvents; 2) the zero valent iron acts as an oxygen scavenger to maintain favorable anaerobic conditions for biological dechlorination; 3) in an anaerobic environment the corrosion of zero valent iron results in the generation of hydrogen which is also favorable to indigenous anaerobic bacteria; and 4) the addition of the zero valent iron to Rice's solution reduces the amount of pyruvate, lactate, formate, propionate and acetate that needs to be provided as the zero valent iron also serves as an electron donor. *Id.*, ¶ 111. Given the well-known use of zero valent iron and the well-known use of organic electron donors individually for the dechlorination of chlorinated solvents in the soil, as well as the known symbiotic characteristics of the two methods to one another, a POSA would have been confident in the success of combining the teachings of Rice and Cantrell as described above. Id., ¶ 112.

Therefore, it would have been obvious to one skilled in the art to add the mixture including a zero valent metal as taught by Cantrell to the solution used in the dechlorination method described by Rice for the reasons listed above with a high likelihood of success Id., ¶ 113.

The zero valent iron in the mixture undergoes many reactions in the subsurface, including reactions with the dissolved chlorinated solvents in the groundwater. Id., ¶ 114 [1.2]. The addition of zero valent iron to the subsurface that is under reducing conditions (anaerobic) will result in the formation of H<sub>2</sub> gas as known in the art. *Id.*, ¶ 114 [1.2]. This hydrogen gas enhances the anaerobic biological degradation process of the residual dissolved chlorinated solvents by the indigenous anaerobic bacteria. *Id.*, ¶ 114 [1.2]. The mixture also contains suitable anaerobic microorganisms to remove target contaminants and likewise promotes biological reactions involving chlorinated solvents in the soil. Id., ¶ 114 [1.2]. As described by Rice, supplying lactate, propionate and/or acetate (organic hydrogen donors) stimulates the dechlorination process by providing electrons that allow indigenous anaerobic bacteria to biologically degrade residual chlorinated solvents. *Id.*, ¶ 114 [1.2].

### **b.** Claim 14 the method of Claim 1, wherein said metal is iron.

Cantrell discloses the metal is zero valent iron. PXY-1011, ¶ 114 [14.0]. See VII.E.3.a.ii (above).

<u>c.</u> <u>Claims 2, 5</u> The method of claim 1 further including the step of supplying a reducing agent into said soil pathways to remove oxygen from groundwater and soil moisture and The method of claim 2 wherein said reducing agent is sodium sulfite.

Rice describes the addition of a reducing agent/deoxygenating agent, specifically sodium sulfite, into the aqueous solution to deoxygenate water. PXY-1006, 3:47-57, Claims 1, 3, and 4; PXY-1011, ¶ 114 [2.0], [5.0]. The aqueous solution is injected into the soil. PXY-1006, Abstract, 4: 9-10, Claim 1; PXY-1011, ¶ 114 [2.0].

The water of the aqueous solution can be deoxygenated groundwater or can be water that is physically or chemically deoxygenated, for example, [...] by adding a strong reducing agent, such as, but not limited to, sulfite or citrate. Sodium sulfite at approximately 8 to 12 mg/L per mg/L dissolved oxygen is suitable. PXY-1006, 3:47-53.

<u>d. Claim 3</u> the method of Claim 1, wherein the steps of supplying said mixture and said organic hydrogen donor are carried out by placing an injection rod into the soil and then injecting them under pressure through an injection rod. Rice describes the injection of the solution into the ground under pressure.

PXY-1011, ¶ 114 [3.0].

The injection system can comprise a conventional well of sufficient depth to ensure that the desired treatment zone is established. It is desirable to inject the solution as quickly and as uniformly as possible, keeping in mind the limits imposed by the hydraulic conductivity at the injection site. The time required to produce a zone of influence of desired radius and volume is readily determined from the volume and the injection rate. The solution can flow into the injection system, or, to reduce the injection time, the solution can be injected by regulated flow under a positive pressure. PXY-1006, 3:35-46.

One skilled in the art would understand the injection system with a regulated flow under a positive pressure would necessarily include a pipe/line/tube, etc. to inject the solution into the injection well in the ground. PXY-1011, ¶ 114 [3.0].

### <u>e. Claim 7</u> the method of claim 1 wherein the mixture further includes nutrients.

Rice describes and claims the addition of nutrients to support the growth of microorganisms to enhance dechlorination. PXY-1006, 1:37-39, 4:28-43, Claim 2; PXY-1011, ¶ 114 [7.0].

It is generally expected that most sites to be remediated contain indigenous anaerobic microorganisms, such as bacteria, that can reductively dehalogenate the halogenated organic compound, if reducing conditions are established. However, if anaerobic microorganisms are not indigenous to the site, suitable anaerobic microorganisms can be provided, either directly into the site or in the injected aqueous solution. For example, bacteria classified as Desulfomonile, Dehalospirilum, and Dehalobacter (in particular, Desulfomonile tiedjei, Dehalospirilum multivorans and Dehalobacter restrictus) are examples of microorganisms that can reductively dehalogenate organic compounds. If the site does not generally support growth of the microorganisms, additional nutrients can be added to support the added microorganisms. PXY-1006, 4:28-43.

PCE degradation can be enhanced by adding trace nutrients, and an electron donor substrate such as lactate, under reducing conditions. PXY-1006, 1:37-39

2. A method as claimed in claim 1 wherein the solution further comprises nutrients required for growth of the anaerobic microorganism. PXY-1006, Claim 2.

### <u>f. Claim 15</u> The method of claim 1 wherein said metal is in a colloidal suspension.

Cantrell discloses a zero valent iron is introduced into the soil as a colloidal suspension. *See VII.E.3 a.ii* (above); PXY 1011, ¶ 114 [15.0].

### <u>g. Claim 16</u> The method of claim 15 wherein the colloidal suspension includes a reducing agent.

As described above (*See VII.E.3.c*), the aqueous solution of Rice which forms the fluid of the colloidal suspension contains sodium sulfite. PXY-1006,

3:47-57; PXY-1007, 4:49-64,11:31-32, 18:3-6; PXY-1011, ¶ 114 [16.0]. Sodium sulfite is a reducing agent. PXY-1006, 3:47-57; PXY-1011, ¶ 114 [16.0].

### <u>h.</u> <u>Claim 11</u> The method of claim 1 wherein said mixture including a zero valent metal is a colloidal suspension in a sodium sulfite solution;

As described above (*See VII.E.3.a, c*), the aqueous solution of Rice which forms the colloidal suspension with iron contains sodium sulfite. PXY-1011, ¶ 114 [11.0].

### <u>i. Claim 12</u> The method of claim 3 further including, after the step of injecting the organic hydrogen donor, an additional step of injecting into the soil a sodium sulfite and nutrient solution to provide for further in-situ mixing and penetration of anaerobic stimulating products.

Rice discloses a method of injecting sodium sulfite and nutrient solution into the soil. PXY-1006, 1:37-39, 3:47-57, 4:28-43, Claim 2; PXY-1011, ¶ 114 [12.0]. Rice describes "repeated applications of the method may be indicated" and thus a subsequent application of the mixture would meet the additional step of injecting sodium sulfite and nutrient solution after an earlier injection of the organic hydrogen donor. PXY-1006, 4:54-60; PXY-1011, ¶ 114 [12.0].

### G. Ground 5: Claims 4, 10 and 13 are unpatentable under 35 U.S.C. § 103(a) over Vance in view of Schuring

#### 1. The Schuring reference

U.S. Patent No. 5,032,042 to Schuring et al., titled "METHOD AND APPARATUS FOR ELIMINATING NON-NATURALLY OCCURRING SUBSURFACE, LIQUID TOXIC CONTAMINANTS FROM SOIL" filed on June 26, 1990 and issued on July 16, 1991, qualifies as prior art under 35 U.S.C. § 102(b) ("Schuring") (attached herein as Exhibit PXY-1024).

Schuring describes pneumatic fracturing to increase soil permeability, recognizing "the effectiveness of virtually all in situ treatment processes in the vadose zone can be enhanced by increasing the permeability of the soil formation." PXY-1024, 4:50-53, 9:12-38; PXY-1011, ¶ 124-126.

### 2. Detailed Analysis of Claims 4, 10 and 13 in view of Vance and Schuring

<u>a. Claim 4</u> The method of claim 3 further including the preliminary step of injecting a gas under pressure through said injection rod and into said soil to establish preferential delivery pathways therein.

Vance discloses the limitation of Claim 3 (*see VII.C.2.c*); however Vance does not expressly disclose a preliminary step of injecting the nitrogen gas as recited in Claim 4. Schuring, however, describes increasing the effectiveness of in-situ bioremediation processes by pneumatic fracturing prior to treatment.

Schuring describes pneumatic fracturing "introduces flow channels into the soil" which enables distribution of the remediation treatments. PXY-1024, 5:32, 9:16-26; PXY-1011, ¶ 132 [4.0].

It would have been obvious to a POSA to modify the method of Vance by injecting nitrogen gas under pressure into the soil prior to applying the treatment as taught by Schuring. A POSA would have multiple reasons to make the modification, for example: 1) as noted by Schuring, the modification would result in greater permeability which enhances the effectiveness of in situ treatment processes; 2) the modification results in better distribution of the materials within the treatment zone; 3) the modification would open up additional soil pathways within the treatment zone; 4) the modification would allow for an increased rate of delivery of the slurry of iron and corn syrup; 5) would result in a dispersal of the slurry within the subsurface over significant horizontal distances; and 6) the modification would reduce the number of injection wells required. PXY-1011, ¶ 130.

### b. Claim 10 The method of claim 4 wherein said gas is from the group of nitrogen and carbon dioxide.

The pneumatic fracturing taught by Schuring utilizes air as the gas, and air comprises nitrogen and carbon dioxide. PXY-1024, 8:66-9:1; PXY-1011, ¶ 132 [10.0]. Moreover, given the method of Vance utilizes nitrogen gas to inject the

mixture, it would have been obvious to a POSA to utilize nitrogen as the fracturing gas. A POSA would have many reasons to use nitrogen, for example: 1) the use of nitrogen would maintain an anaerobic environment conducive to reductive dechlorination taught by Vance; and 2) the system of Vance already utilizes nitrogen and thus a separate pneumatic system would not be required. PXY-1011, ¶ 132 [10.0].

## <u>c. Claim 13</u> The method of claim 4 further including a final step of gas injection to clear said injection rod and fluid conduit lines connected thereto.

Neither Schuring nor Vance discloses purging the injection rod or connected lines as a final step. Cleaning equipment after use is well known in the art and has been practiced for millennia. Likewise, ensuring all the remediation treatment is supplied into the soil is economic common sense. PXY-1011, ¶ 132 [13.0]. It is further understood by a POSA that purging the injection rod and conduit lines with the injection of gas is a well-known and successful cleaning method. *Id.*, ¶ 132 [13.0].

It would have been obvious to a POSA to add as a final step to the method of Vance and Schuring as described in Claim 4, the injection of gas into the corn syrup and iron delivery system of Vance to purge any remaining slurry. PXY-1011, ¶ 132 [13.0]. A POSA would be motivated to add the step to clean the

equipment and supply all the slurry into the soil requiring remediation. PXY-1011, ¶ 132 [13.0].

### H. Ground 6: Claim 8 is unpatentable under 35 U.S.C. § 103(a) over Hitchens in view of Grasso

<u>Claim 8</u> The method of claim 7 wherein said nutrients are organic ammonia and ortho-phosphate.

Hitchens discloses providing nutrients in the mixture as described in claim 7. *See VII.B.2.a* (above). Hitchens does not expressly describe the nutrients are ammonia and orthophosphate. However, Grasso describes introducing nutrients such as ammonia and orthophosphate can "enhance the rate and extent of biodegration." PXY-1014, 5.1.4.1; PXY-1011, ¶ 135, 138 [8.0].

Phosphorus is added in the form of inorganic orthophosphate. PXY-1014, 5.1.4.1.

Nitrogen can be introduced in the nutrient media in either inorganic or organic forms. The most common inorganic forms are as ammonium or nitrate ions. *Id.*, 5.1.4.1.

When nitrate is supplied as the only nitrogen source, it must be first reduced to ammonia before the bacterium can use it. *Id.*, 5.1.4.1.

It would have been obvious to a POSA to include ammonia and orthophosphate as taught by Grasso, as the nutrients injected into the soil as taught by Hitchens. A POSA would have many reasons to include ammonia and orthophosphate as nutrients, for example: 1) the application of such nutrients can enhance the rate of and extent of biodegradation; and 2) nitrogen and phosphorus are the limiting factor in the subsurface environment and must be supplied to ensure biodegradation. PXY-1011, ¶ 136.

### I. Ground 7: Claim 6 is unpatentable under 35 U.S.C. § 103(a) over Hitchens in view of Odom

# Claim 6The method of claim 1 wherein said organic<br/>hydrogen donor further includes vitamins B2<br/>and B12.

Hitchens discloses the method of Claim 1 as shown above (*See VII.B.2.a*). Hitchens, while recognizing the dechlorination properties of B12, does not disclose supplying B2 and B12 into the soil. PXY-1003, 2:63-67; PXY-1011, ¶¶ 140-141. Odom, however, discloses that the introduction of B2 and B12 together greatly accelerated dechlorination. PXY-1025, Abstract, 16:7-10; PXY-1011, ¶ 143, 148 [6.0].

The invention relates to a method for the dehalogenation or organohalogen compounds commonly found as pollutants in soil and ground water. The instant method utilized anaerobic bacteria comprising an extracellular electron transfer protein coupled with a tetrapyrrole catalyst to effect the reductive dehalogenation of halogenated organics. PXY-1025, Abstract.

The reagents will be introduced at the injection well in the same manner as the nutrients. PXY-1011, ¶ 145.

Dechlorination at levels 30-100 fold higher than hydrogenase-C3 were observed when both riboflavin and the tetrapyrrole were present. PXY-1025, 16:7-10.

Riboflavin is vitamin B2 and the tetrapyrrole in Odom is described as cyanocoblalamin or vitamin B12. PXY-1025, 15:30-16:15; PXY-1011, ¶ 144.

It would have been obvious for a POSA to modify the method of Hitchens by supplying vitamins B2 and B12 as taught by Odom along with the organic hydrogen donor into the sub soil for dechlorination. PXY-1011, ¶ 146. A POSA would be motivated to make a modification to increase the amount of dehalogenation/dechlorination by several fold as taught by Odom as well as increasing the rate of dehalogenation/dechlorination. PXY-1011, ¶ 146.

#### **VIII.** Conclusion

For the reasons set forth above, Petitioner has established a reasonable likelihood of prevailing with respect to at least one of the claims of the '709 Patent and asks that the Patent Office order an *inter partes* review, and find that the Challenged Claims are obvious over the prior art references identified above.

Respectfully submitted,

/s/ D. Joseph English D. Joseph English Duane Morris LLP 505 9th Street NW Washington, D.C. 20004 11/24/2015

Dated:

#### **CERTIFICATION OF SERVICE ON PATENT OWNER**

Pursuant to 37 C.F.R. §§ 42.6(e), 42.8(b)(4) and 42.105, the undersigned certifies that on the 24th day of November 2015, a complete and entire copy of this Petition for *Inter Partes* Review of U.S. Patent No. 7,531,709 and all supporting exhibits were served via Federal Express, postage prepaid to the following correspondence addresses:

Gavin P. Lentz (Pa. 53609) Bryan R. Lentz (Pa. 71383) BOCHETTO & LENTZ PC 1524 Locust Street Philadelphia, PA 19102

Douglas J. Ryder (Pa. 87281) Ryder, Lu, Mazzeo & Konieczny LLC 808 Bethlehem Pike, Suite 200 Colmar, PA 18915

and by Express Mail<sup>®</sup>, postage prepaid to the address of record for the '709 patent to the following correspondence address:

Ryder, Lu, Mazzeo & Konieczny, LLC P.O. Box 670 Plymouth Meeting PA 19462

> /D. Joseph English/ D. Joseph English, Reg. No. 42,514 505 9th Street, N.W., Suite 1000 Washington, D.C. 20004 P: (202) 776-7800 F: (202) 776-7801 DJEnglishn@duanemorris.com

ATTORNEY FOR PETITIONER