

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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BEFORE THE PATENT TRIAL AND APPEAL BOARD

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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

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Inter Partes Review No.: Unassigned

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**DECLARATION OF DR. JOHN A. BERGENDAHL IN SUPPORT OF  
PETITION FOR INTER PARTES REVIEW OF  
U.S. PATENT NO. 7,531,709**

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## **I. Introduction**

1. I am over the age of eighteen (18) and otherwise competent to make this declaration.

2. I have been engaged as an expert witness on behalf of Petitioner PeroxyChem LLC (“Petitioner” or “PeroxyChem”) in the above-captioned Petition for *Inter Partes* Review (“the Petition”). I am being compensated for my time in connection with this matter at my standard consulting rate, which is \$200 per hour. My compensation is in no way dependent on the outcome of this *inter partes* review.

3. This Petition for *inter partes* review involves U.S. Patent No. 7,531,709 (“the ‘709 Patent”) (PXY-1001). The ‘709 Patent is entitled “METHOD FOR ACCELERATED DECHLORINATION OF MATTER,” and lists Michael Scalzi and Wade Meese as the inventors. The ‘709 Patent issued with Claims 1-16 (the “Challenged Claims”).

4. The ‘709 Patent issued May 12, 2009, from U.S. Patent Application No. 11/423,749 which was filed June 13, 2006 (“the ‘749 Application”) (Exhibit PXY-1008). The ‘709 Patent claims priority to U.S. Patent Application No. 10/610,558 filed July 2, 2003 (“the ‘558 Application”) (Exhibit PXY-1023) which is now issued as U.S. Patent No. 7,129,388 (“the Parent Patent”). The ‘709 Patent

additionally claims priority to U.S. Provisional Patent Application No. 60/437,983 filed January 6, 2003 (“the ‘983 Application”) (PXY-1009).

5. For the purposes of this *inter partes* review, I have been instructed to assume that the effective filing date of the Claims of the ‘709 Patent challenged by the Petitioner in this *inter partes* review is the earliest possible priority date, i.e., the January 6, 2003, filing date of U.S. Provisional Patent Application No. 60/437,983.

6. I understand that according to USPTO records, the ‘709 Patent is currently assigned to Innovative Environmental Technologies, Inc. (“Innovative Environmental Technologies” or “Patent Owner”).

7. The ‘709 Patent is directed to “an accelerated dechlorination of subsurface matter by anaerobic microorganisms in conjunction with oxygen scavengers, vitamins, nutrients, and zero valent metals.” PXY-1001, 1:17-21. I am familiar with the technology described in the ‘709 Patent as of the earliest possible priority date of January 6, 2003.

8. In preparing this Declaration, I have reviewed the ‘709 Patent (PXY-1001), the file history of the ‘709 Patent (PXY-1002), and each of the documents cited herein, and I have considered these documents in light of the general knowledge in the art as of January 6, 2003. In formulating my opinions, I have relied upon my experience in the relevant art. I have also considered the viewpoint

of a person of ordinary skill in the art (“POSA”) in the field of groundwater or soil contaminant treatment, as of January 6, 2003.

9. I have been asked to provide my technical expertise, analysis, insights and opinions regarding the ‘709 Patent and relevant references that form the basis of the grounds of rejection set forth in the accompanying Petition for *inter partes* review of the ‘709 Patent. As described in detail below, I offer the following opinions in this Declaration:

- A POSA would have found Claims 1, 7, 9, and 14 of the ‘709 Patent to be anticipated by U.S. Patent No. 6,265,205 to Hitchens et al. (“Hitchens”) (PXY-1003). Hitchens teaches each element of Claims 1, 7, 9, and 14 to a POSA.
- A POSA would have found Claims 1, 3, 7, 9, and 14-16 of the ‘709 Patent to be anticipated by U.S. Patent Application Publication No. 2002/0151602 to Vance et al. (“Vance”) (PXY-1010). Vance teaches each element of Claims 1, 3, 7, 9, and 14-16 to a POSA.
- A POSA would have found Claims 1, 3, 7, and 14 of the ‘709 Patent to be anticipated by PCT Published Application WO 98/49106 to Alvarez et al. (“Alvarez”) (PXY-1004). Alvarez teaches each element of Claims 1, 3, 7, and 14 to a POSA.

- A POSA would have found Claims 1-3, 5, 7, 11, 12, and 14-16 of the ‘709 Patent to be obvious over U.S. Patent No. 6,001,252 to Rice et al. (“Rice”) (PXY-1006) in view of U.S. Patent No. 5,857,810 to Cantrell et al. (“Cantrell”). Rice in view of Cantrell teaches each element of Claims 1-3, 5, 7, 11, 12, and 14-16 to a POSA, and a POSA would have been motivated to combine the teachings of these references.
- A POSA would have found Claims 4, 10, and 13 of the ‘709 Patent to be obvious over Vance in view of U.S. Patent No. 5,032,042 to Schuring et al. (“Schuring”) (PXY-1024). Vance in view of Schuring teaches each element of Claims 4, 10, and 13 to a POSA, and a POSA would have been motivated to combine the teachings of these references.
- A POSA would have found Claim 8 of the ‘709 Patent to be obvious over Hitchens in view of “Hazardous Waste Site Remediation” by Domenic Grasso (“Grasso”) (PXY-1014). Hitchens in view of Grasso teaches each element of Claim 8 to a POSA, and a POSA would have been motivated to combine the teachings of these references.
- A POSA would have found Claim 6 of the ‘709 Patent to be obvious over Hitchens in view of U.S. Patent No. 5,587,317 to Odom et al.

(“Odom”) (PXY-1025). Hitchens in view of Odom teaches each element of Claim 6 to a POSA, and a POSA would have been motivated to combine the teachings of these references.

10. In summary and as will be explained in further detail below, the claims of ‘709 Patent are known, as are their functions and predictable results. 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.

## **II. List of Documents I Considered in Forming My Opinions**

11. In formulating my opinions, I have considered the following documents:

<b>Exhibit Number</b>	<b>Exhibit Title</b>
PXY-1001	U.S. Patent Number 7,531,709 Patent (the “‘709 Patent”)
PXY-1002	U.S. Patent Number 7,531,709 File History
PXY-1003	U.S. Patent Number 6,265,205 Patent (“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”)



<b>Exhibit Number</b>	<b>Exhibit Title</b>
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-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. p.3448-3454. “DDT, DDD and DDE Dechlorination by Zero-Valent Iron,” 1997 (“Sayles”)
PXY-1014	Domenic 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.

<b>Exhibit Number</b>	<b>Exhibit Title</b>
	("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) ("Rittman")
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")

### **III. My Background and Qualifications**

12. My expertise qualifies me to do the type of analysis required in this case for the field of groundwater or soil contaminant treatment. Of particular relevance, I am an associate professor in the Department of Civil and Environmental Engineering at Worcester Polytechnic Institute, and I am currently the Director of the Undergraduate Environmental Engineering Program. At Worcester Polytechnic Institute, I have developed and teach courses related to treatment of contamination including: CE3060 Water Treatment, CE3061 Wastewater Treatment, CE4600 Hazardous and Industrial Waste Management, ES2800 Environmental Impacts of Engineering Decisions, CE561 Advanced

Principles of Wastewater Treatment, CE569 Environmental Treatability Laboratory, and CE572 Physical and Chemical Treatment Processes.

13. Throughout the remainder of this Declaration, I will refer to the field of groundwater or soil contaminant treatment, as the “relevant field” or the “relevant art.” In formulating my opinions, I have relied upon my training, knowledge, and experience in the relevant art. A copy of my current curriculum vitae is provided as PXY-1027, and it provides a comprehensive description of my academic and employment history, including a list of relevant art publications for at least the last 20 years.

14. I started in the relevant art as a student at the University of Connecticut (“UCONN”) where I earned a B.S. in Mechanical Engineering in 1985, an M.S. in Environmental Engineering in 1996, and a Ph.D. in Chemical Engineering in 1999. Relevant coursework that I took during my studies included: Environmental Engineering Chemistry, Environmental Organic Chemistry, Bioprocesses, Contaminant Source Remediation, Contaminant Transport, Chemical Engineering Thermodynamics, Transport Phenomena, and Reaction Kinetics.

15. In 1985, I began my career in engineering when I joined General Dynamics, Electric Boat Division, Groton, CT as an Associate Engineer (“EB”). Between 1985 and 1992, I continued my career at EB serving as an Associate

Engineer, Engineer, and Senior Engineer. After leaving EB, I had the privilege of serving during an internship in the Technical Assistance Program at the Connecticut Hazardous Waste Management Service in Hartford, CT.

16. Throughout my doctoral program I served as a Teaching Assistant and a Research Associate. Following completion of my Ph.D., I served as a Postdoctoral Research Fellow with the Environmental and Water Resources Engineering Program for the Department of Civil Engineering, at the University of Texas, Austin. In January 2000 I joined the faculty at Worcester Polytechnic Institute (“WPI”) in Worcester, MA as an Assistant Professor. I am currently a tenured member of the faculty at WPI as an Associate Professor, serving in that role since July 2006.

17. During my tenure with the faculty at WPI, I have instructed undergraduate and graduate level courses in Physical and Chemical Treatment Processes, Advanced Principles of Wastewater Treatment, Civil Engineering and Computer Fundamentals, Environmental Treatability Laboratory, Wastewater Treatment, and Treatment System Hydraulics.

18. Throughout a career spanning over two decades, including fifteen years in research and teaching at WPI, I have published numerous articles and contributed to scholarly works devoted to environmental remediation, including, for example, peer-reviewed publications such as “Biodegradability of Fluorinated

Fire-Fighting Foams in Water,” by Bourgeois, Bergendahl, and Rangwala, published in 2015 in *Chemosphere*, 131, 104-109, “An Equilibrium Study on Trichloroethylene Adsorption and Desorption from ZSM-5 in Water,” by Hawley, Bergendahl and Thompson, published in 2013 in *Energy and Environmental Engineering*, 1, 5- 9, “A Kinetic Model for the Aqueous Degradation of MTBE by  $\text{Fe}^0/\text{H}_2\text{O}_2$ ” by Al Ananzeh, Bergendahl and Thompson, published in 2007 in *Progress in Reaction Kinetics and Mechanism*, 32, 131-151, “Kinetic Model for the Degradation of MTBE by Fenton’s Oxidation” by Al Ananzeh, Bergendahl, and Thompson published in 2006 in *Environmental Chemistry*, 3, 40-47, “Fenton’s Oxidation of MTBE with Zero-Valent Iron” by Thies and Bergendahl published in 2004 in *Water Research*, 38, 327-334, and “Pilot-Scale Fenton’s Oxidation of Organic Contaminants in Groundwater Using Autochthonous Iron” by Hubbard, Bergendahl and Grasso published in 2003 in *Journal of Hazardous Materials*, 99, 43-56, 2003.

19. I authored a book chapter “Environmental Issues of Gasoline Additives – Aqueous Solubility and Spills” in *Solubility, Thermodynamics and Environmental Issues*, edited by T. Letcher, published by Elsevier in 2007. I also authored a textbook *Treatment System Hydraulics*, published by American Society of Civil Engineers in 2007, and am currently writing a book *Environmental Impacts of Engineering Decisions*, which is to be published by Wiley-Blackwell.

20. I am currently a member of the American Institute of Chemical Engineers, American Society of Civil Engineers, and the Association of Environmental Engineering and Science Professors. I have been on the Editorial Board of *Soil and Sediment Contamination, An International Journal*, since 2003, and have reviewed manuscripts for many professional journals (including *Environmental Science and Technology*, *Environmental Engineering Science*, etc.), and have served on proposal review panels for organizations such as the *Environmental Protection Agency*, the *Transportation Research Board*, ACS's *Petroleum Research Fund*, and the *National Science Foundation*. I am a licensed Professional Engineer in Connecticut.

**IV. Person of Ordinary Skill in the Art (POSA)**

21. I am familiar with the knowledge and capabilities of one of ordinary skill in the art. For example, my work as a registered professional engineer with doctoral level studies in chemical and environmental engineering and research interests focused on physical and chemical treatment processes, including chemical oxidation of organics in water, absorption of organic contaminants onto activated carbon and novel adsorbents during that period, allowed me to become personally familiar with the level of skill of individuals and the general state of the art. Unless otherwise stated, my testimony below refers to the knowledge of one of

ordinary skill in the art as of January 6, 2003, the earliest possible effective filing date of the '709 Patent.

22. I have been informed and understand that 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.

23. 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.

24. 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.

## **V. Relevant Legal Standards**

25. I am not a lawyer and will not provide any legal opinions. Although I am not a lawyer, I have been informed and understand that certain legal standards

are to be applied by technical experts in forming opinions regarding the meaning and validity of patent claims. I have been asked to provide my opinions regarding whether the claims of the '709 Patent are anticipated or would have been obvious to a person having ordinary skill in the art at the time of the alleged invention, in light of the prior art.

26. I have been informed and understand that, to anticipate a claim under 35 U.S.C. § 102, a reference must teach every element of the claim either expressly or inherently to a person having ordinary skill in the relevant art.

27. Further, I have been informed and understand that a patent claim is not patentable under 35 U.S.C. § 103 if the differences between the patent claim and the prior art are such that the claimed subject matter as a whole would have been obvious at the time the claimed invention was made to a person having ordinary skill in the relevant art. Obviousness, as I have been informed and understand, is based on the scope and content of the prior art, the differences between the prior art and the claim, the level of ordinary skill in the art, and, to the extent that they exist, certain objective indicia of non-obviousness.

28. I understand that objective indicia can be important evidence regarding whether a patent is obvious or nonobvious, if it has an appropriate nexus to the claimed invention, i.e., is a result of the merits of a claimed invention (rather than the result of design needs or market-pressure advertising or similar activities).



Such indicia include: commercial success of products covered by the patent claims; a long-felt need for the invention; failed attempts by others to make the invention; copying of the invention by others in the field; unexpected results achieved by the invention as compared to the closest prior art; praise of the invention by the infringer or others in the field; the taking of licenses under the patent by others; expressions of surprise by experts and those skilled in the art at the making of the invention; and the patentee proceeded contrary to the accepted wisdom of the prior art.

29. I have been informed that whether there are any relevant differences between the prior art and the claimed invention is to be analyzed from the view of a person of ordinary skill in the relevant art at the time of the invention. As such, my opinions below as to a person of ordinary skill in the art are as of the time of the invention, even if not expressly stated as such; for example, even if stated in the present tense.

30. In analyzing the relevance of the differences between the claimed invention and the prior art, I have been informed that I must consider the impact, if any, of such differences on the obviousness or non-obviousness of the invention as a whole, not merely some portion of it. The person of ordinary skill faced with a problem is able to apply his or her experience and ability to solve the problem and also look to any available prior art to help solve the problem.

31. I have been informed that a precise teaching in the prior art directed to the subject matter of the claimed invention is not needed. I have been informed that one may take into account the inferences and creative steps that a person of ordinary skill in the art would have employed in reviewing the prior art at the time of the invention. For example, if the claimed invention combined elements known in the prior art and the combination yielded results that were predictable to a person of ordinary skill in the art at the time of the invention, then this evidence would make it more likely that the claim was obvious. On the other hand, if the combination of known elements yielded unexpected or unpredictable results, or if the prior art teaches away from combining the known elements, then this evidence would make it more likely that the claim that successfully combined those elements was not obvious.

32. I have been informed and understand that there are recognized, exemplary, rationales for combining or modifying references to show obviousness of claimed subject matter. Some of the rationales include the following: combining prior art elements according to known methods to yield predictable results; simple substitution of one known element for another to yield predictable results; use of a known technique to improve a similar device (method or product) in the same way; applying a known technique to a known device (method or product) ready for improvement to yield predictable results; choosing from a finite

number of identified, predictable solutions, with a reasonable expectation of success; known work in one field of endeavor may prompt variations of it for use in either the same field or a different one based on design incentives or other market forces if the variations are predictable to one of ordinary skill in the art; and some teaching, suggestion, or motivation in the prior art that would have led one of ordinary skill to modify the prior art reference or to combine prior art teachings to arrive at the claimed invention.

## **VI. The ‘709 Patent**

### **A. Description**

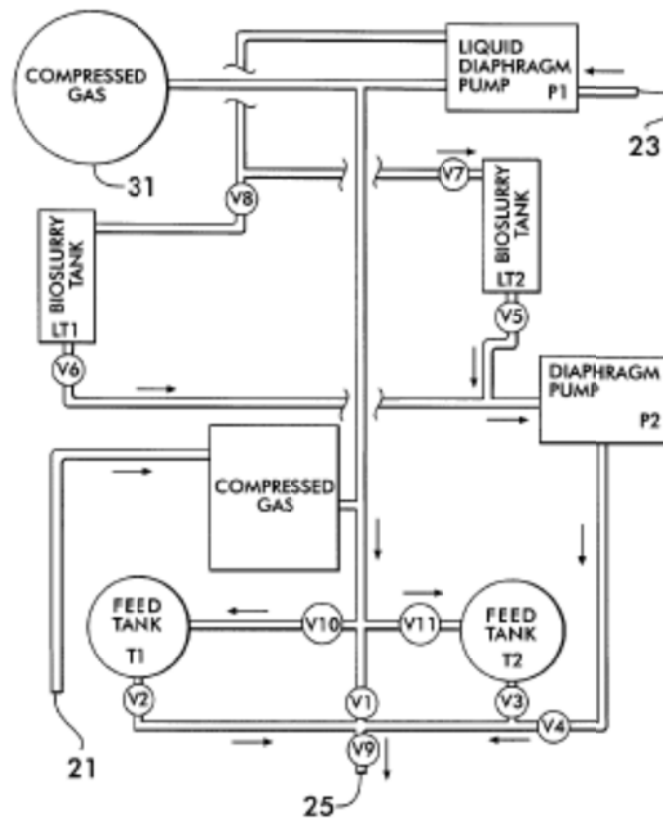
33. The ‘709 Patent describes and claims a method for in situ soil remediation by supplying a first and a second remediation element into the soil so that naturally occurring processes dechlorinate/dehalogenate contaminants in the soil. Specifically, the ‘709 Patent describes and claims a method for 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.

34. 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.

35. The ‘709 Patent discloses 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.

36. The only figure of the ‘709 Patent illustrates a preferred delivery system. PXY-1001, 4:46-47.



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

#### Step 1: Subsurface Pathway Development

Pathways for delivery of remediation substances are ensured with gas injection.

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

A solution of sodium sulfite and nutrients (nitrogen and ortho-phosphate) is immediately injected into the subsurface pathways and voids that were developed during the gas injection step. Sodium sulfite acts as an oxygen scavenger, iron reducer and sulfate source ....

### Step 3: Zero Valent Metal Injection

Immediately following the sodium sulfite/bioslurry solution injection, a colloidal suspension of a metal powder is added to an additional quantity of the bioslurry solution and the colloidal suspension is injected to reduce concentrations of dissolved-phase CVOCs while providing for rapidly generated hydrogen, the evolution of hydroxides and as a result overall microbial stimulation and biofilm formation.

### Step 4: Anaerobic Hydrogen Source Injection

An anaerobic organic hydrogen source is injected immediately after the ZVI injection to provide a slow release hydrogen source for the anaerobic dechlorination of the CVOCs. Vitamin B12 and riboflavin B2 is mixed with the anaerobic stimulating hydrogen source to provide essential micro enzymes at the anaerobic sites.

### Step 5: Sodium Sulfite/Nutrient Injection

A second injection of the sulfite/nutrient mixture ...

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

*See* PXY-1001, 2:50 – 3:42.

38. 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.

39. The '709 Patent discloses that the organic hydrogen donor is an electron donor source 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.

40. 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.

41. Claims 1-16 of the '709 Patent 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.

42. Specifically, the Challenged Claims recite:

*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.*

*2. 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.*

*4. 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.*

*5. The method of claim 2 wherein said reducing agent is sodium sulfite.*

*6. The method of claim 1 wherein said organic hydrogen donor further includes vitamins B2 and B12.*

*7. The method of claim 1 wherein the mixture further includes nutrients.*

*8. The method of claim 7 wherein said nutrients are organic ammonia and ortho-phosphate.*



*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.*

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

*11. The method of claim 1 wherein said mixture including a zero valent metal is a colloidal suspension in a sodium sulfite solution.*

*12. 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.*

*13. The method of claim 4 further including a final step of gas injection to clear said injection rod and fluid conduit lines connected thereto.*

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

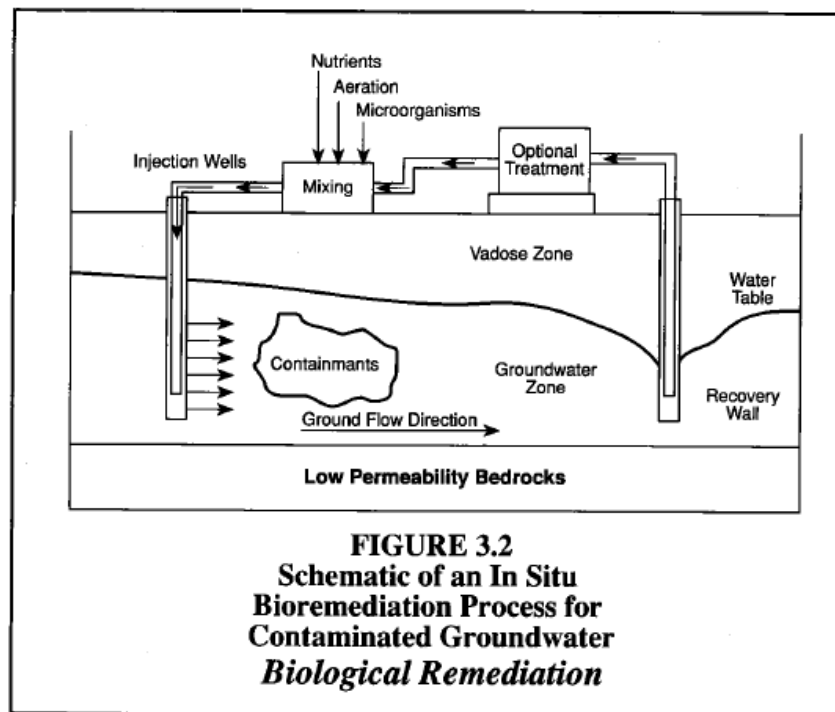
*15. The method of claim 1 wherein said metal is in a colloidal suspension.*

*16. The method of claim 15 wherein the colloidal suspension includes a reducing agent.*

## B. Background of the Relevant Art

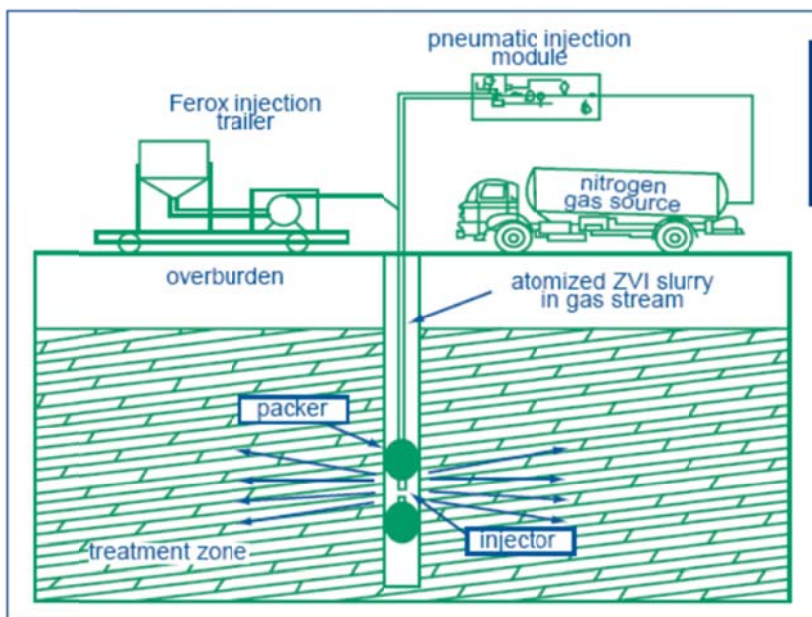
43. As will be further explained in the following paragraphs, as of January 2003 it was known in the art to dechlorinate contaminated soil using zero valent metals and organic hydrogen donors, either alone or in combination. Note that the use of the term “organic hydrogen donor” throughout this declaration is consistent with the assumed definition detailed in section VII.A.

44. Injection and fracturing processes, including the use of injection wells to treat contaminated soil and groundwater had long been known and practiced in the art. As one example, Grasso provides the following schematic of an in situ bioremediation process using injection wells:



PXY-1014, J-12.

45. As another example, TNT describes injecting zero valent iron (ZVI) powder and water through an injection rod in the ground into the treatment zone with pressure. PXY-1005, p. 4. The Ferox process described in TNT employs a driven casing/nozzle system or open borehole to create an in-situ, iron-enriched zone of soil above or below the ground-water table. Using potable water and ZVI powder, the slurry is fed into the nitrogen gas stream, which atomizes the water and iron particles to produce a fine mist. This transformation results in dispersal of the iron powder within the subsurface over significant horizontal distances using relatively low injection pressures (Figure 3). *Id.* By applying the injections in 3-ft intervals using a down hole packer system, a total of 125 slurry injection events occurred at pressures ranging from 60 to 120 psig. *Id.* The apparatus disclosed by TNT, including the injection rod, is shown in Figure 3 below.



*Figure 3. The injected Ferox<sup>SM</sup> process slurry produces a fine mist that disperses iron powder within the subsurface.*

PXY-1005, Figure 3.

46. Day describes fracturing processes to create channels for iron slurry.

PXY-1012, pp. 6-7.

47. As discussed above, Claim 1 of the '709 Patent is directed to methods for dechlorination of subsoil matter by the elements of supplying into soil pathways: (1) a mixture including zero valent metal; and (2) an organic hydrogen donor. These two elements are found in the prior art in combination (Vance, Hitchens, Alvarez, TNT, Day, Igawa, Cantrell) and individually (Rice, Batchelor, Sayles, Kawabata). Table 1 below provides a helpful summary of the state of the art as of the priority date of the '709 Patent. Table 1 shows that methods of dechlorination were known in the art that supplied (1) a mixture including zero valent metal and (2) an organic hydrogen donor to soil pathways.

<b>'709 Patent Dechlorination Method</b>		
<b>Claim 1</b>	<b>Element (1): Supply Zero Valent Metal</b> <i>Disclosed examples:</i> Zero Valent Iron (Fe <sup>(0)</sup> ), Iron Powder. <i>PXY-1001, 5:41-59; 8:1</i>	<b>Element (2): Supply Organic Hydrogen Donor</b> <i>Disclosed examples:</i> Glucose, Sucrose, Sugars, Alcohols, Chitin, Propionates, Lactates, Acetates, Butyrate. <i>PXY-1001, 4:29-32, 6:57-60.</i>
<b>Prior Art References</b>		
TNT 2002	Zero Valent Iron (Fe <sup>(0)</sup> ) <i>PXY-1005, pp. 2-3</i>	Organic Mulch, Organic Carbon <i>PXY-1005, pp. 1-2</i>
Vance 2000	Zero Valent Iron (Fe <sup>(0)</sup> ) <i>PXY-1010, ¶¶ [118-120]</i>	Corn Syrup (sugar) <i>PXY-1010, ¶¶ [118-120]</i>
Igawa 1999	Fine Iron Particles <i>PXY-1015, Abstract</i>	Sucrose, Glucose <i>PXY-1015, 5:10-21</i>

Day 1999	Granular Iron <i>PXY-1012, p. 3</i>	Guar Gum (sugar) <i>PXY-1012, pp. 5-6</i>
Hitchens 1998	Zero Valent Iron (Fe <sup>(0)</sup> ) <i>PXY-1003, 3:39-56</i>	Glucose, Acetate, Formate, Methanol (Alcohol), Lactate, Propionate, Butyrate <i>PXY-1003, 2:32-44</i>
Rice 1998		Lactate, Propionate And Acetate <i>PXY-1006, 4:9-10</i>
Alvarez 1997	Zero Valent Iron (Fe <sup>(0)</sup> ) <i>PXY-1004, ¶¶ 10, 17</i>	Methanol (Alcohol), Acetate <i>PXY-1004, pp. 23-24, 26</i>
Cantrell 1997	Zero Valent Iron (Fe <sup>(0)</sup> )  <i>PXY-1026, 4:27-35, 5:33-35</i>	Guar Gum (sugar)  <i>PXY-1026, 3:54-60</i>
Sayles 1997	Zero Valent Iron (Fe <sup>(0)</sup> ) <i>PXY-1013, p. 3448</i>	
Batchelor 1995	Zero Valent Iron (Fe <sup>(0)</sup> ) <i>PXY-1007, 4:41-47, claim 1</i>	
Kawabata 1994		Sugar, Chitin <i>PXY-1016, 6:14-20</i>

Table 1. Summary of Prior Art.

48. Dechlorination (or dehalogenation) of contaminants in soil and ground water was accomplished prior to January 2003 through abiotic reduction with an elemental metal. For example, in *In Situ Treatment Technology* the authors discuss research, trials, and field use of zero-valent metals at least as early as 1992. See PXY-1020, 467-470. The authors note that some researchers “have concentrated on using zero-valent iron to dehalogenate chlorinated aliphatic organic compounds” and that such dehalogenation “is an abiotic process.” Id., 467-68. Similarly, in *Environmental Biotechnology: Principles and Applications* authors Bruce E. Rittman and Perry L. McCarty discuss “in situ biobarriers” and note that:

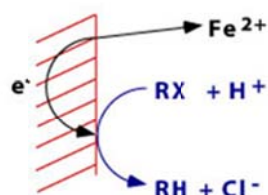
Iron filings also have been used to create an in situ barrier. The oxidation of the elemental iron can bring about reductive dechlorination of solvents

PXY-1021, 718-19.

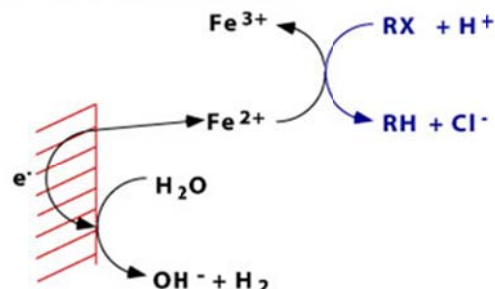
49. Batchelor et al. (“Batchelor”) describes the treatment of contaminated soil with the introduction of elemental metals (zero valent iron), and observes that using iron for the reduction of chlorinated solvents is a known and successful method. PXY-1007, 1:49-64, 2:1-10.

50. As early as 1994 studies confirmed the various pathways for a zero valent metal, such as iron, to effect dechlorination. Matheson and Tratnyek describe three dechlorination pathways: (A) direct reduction, wherein the iron supplies an electron directly to the chlorinated compound; (B) reduction by ferrous iron (i.e. iron in aqueous solution), in which the solubilized iron provides an electron to the chlorinated compound; and (C) reduction by hydrogen, wherein hydrogen produced as a result of anaerobic corrosion of iron performs dechlorination. PXY-1018, 2046.

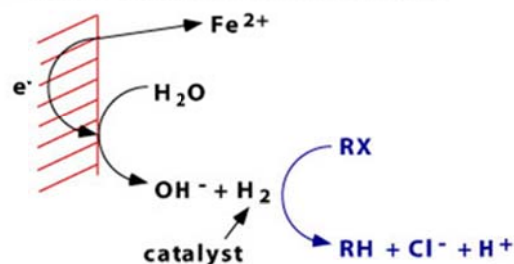
(A) Direct Reduction at the Metal Surface



(B) Reduction by Ferrous Iron



(C) Reduction by Hydrogen with Catalysis



51. Matheson and Tratnyek additionally described their study:

Reduction of chlorinated solvents by fine-grained iron metal was studied in well-mixed anaerobic batch systems in order to help assess the utility of this reaction in remediation of contaminated groundwater. Iron sequentially dehalogenates carbon tetrachloride via chloroform to methylene chloride.

PXY-1018, 2045

52. The use of zero valent metals in dechlorination was in practice in the United States prior to January 2003. 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. Additional examples include the commercialization of zero-valent iron permeable treatment barriers by the University of Waterloo in 1992 by forming EnvironMetal Technologies Inc., a field test demonstration at Canadian Air Force Base Borden, Ontario, from 1991 to 1996, and various site uses tracked by the U.S. Environmental Protection Agency including at the Caldwell Trucking site in New Jersey prior to 1999. PXY-1020, 468-70.

53. 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.

54. 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 on the halogenated organic contaminant (the halogenated organic contaminant as the electron acceptor reacts with an electron donor); and (3) co-metabolism, wherein the metabolizing of a carbon compound results in the conversion of the proximate chlorinated organic compounds (contaminants) to another chemical. PXY-1019, 8.



55. It was further known to use organic hydrogen donors to accelerate these biodegradation processes. PXY-1020, 358-59; *see also* PXY-1014, J-26-27; PXY-1020, 333-36, 340-45, 357-61. Rittman and McCarty describe the “injection of electron donor, electron acceptor, and/or nutrients” to form an in situ biobarrier and note that “[f]ull-scale implementation of this technology for cometabolism of TCE was conducted at Edwards Air Force Base, California.” PXY-1021, 718-19

56. 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. Rice further states “A suitable electron donor substrate is an organic molecule that can be utilized as an energy source by the indigenous anaerobic microorganisms. The electron donor substrate should be a relatively simple organic molecule having available hydrogens ...”. PXY-1006, p. 5.

57. 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.

58. The symbiotic relationship between the use of a zero valent metal and the use of an organic hydrogen donor was also understood prior to January 2003. 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 Sections IX below, Vance describes a dechlorination method of injecting zero valent iron along with a carbohydrate (organic hydrogen donor), specifically corn syrup into the soil. As will be described in further detail in Sections VIII, X, and XI below, Hitchens, Alvarez, Igawa, Cantrell, and Day all describe introducing zero valent metals and organic hydrogen donors for dehalogenation/dechlorination methods for soil remediation.

59. U.S. Patent No. 6,596,190 to Igawa et al. is directed to a “remediation agent for soil contaminated with contaminants such as organic halides” which comprises “a slurry of fine iron particles” and a “hydrophilic binder”. PXY-1015, Abstract, 1:15-16. Igawa further explains that the hydrophilic binder can be an organic hydrogen donor such as sucrose:

The hydrophilic binder covers the surface of the fine iron particles, whereby the surface is protected so as not to be oxidized until the metal halide exhibits reducing action. Examples of the hydrophilic binder include water-soluble resins, for example disaccharides such as sucrose, sucrose derivatives such as higher fatty acid ester of sucrose, monosaccharides such as glucose, alginic acid, pullulan, polyvinyl alcohol (PVA), carboxymethylcellulose (CMC), polyacrylamide, cyamopsis gum, methylcellulose and

hydroxyethylcellulose. Preferred are pullulan (especially because of low viscosity of its aqueous solution), hydroxyethylcellulose, sucrose, glucose and PVA.

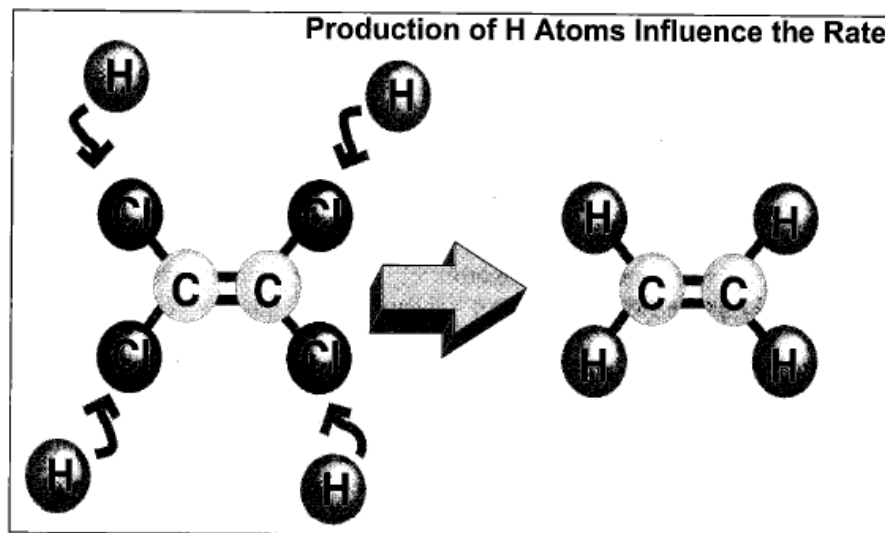
PXY-1015, 9:10-21.

60. In “Geotechnical Techniques for the Construction of Reactive Barriers,” Day et al. disclose a decontamination method “to treat a range of dissolved organic and inorganic compounds including chlorinated solvents.” PXY-1012, p. 2. Day discloses that “the most common [decontamination] technique is to bury granular iron in a trench so that contaminated groundwater passes through the reactive materials, the contaminants are removed and the water becomes ‘clean.’” *Id.* at Abstract. Day further discloses injecting granular iron in combination with “guar gum” as a bio-polymer slurry. “Guar gum is a naturally occurring carbohydrate polymer (combination of nannose and galactose sugars in long linear chains) derived from guar beans.” *Id.* at p. 6.

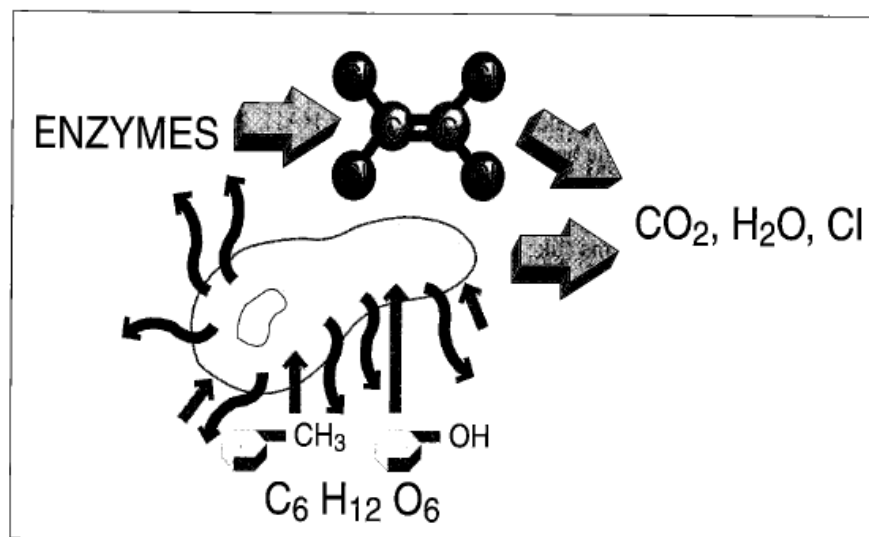
61. *In Situ Treatment Technology* describes the “microbial mechanisms” used to treat Halogenated Aliphatic Hydrocarbons, including the mechanisms claimed in the ’709 Patent namely hydrogenolysis and cometabolic degradation:

As with most microbially mediated processes in the subsurface, the microbial processes that take place in a reactive zone are a collection of processes that result from the natural selection of microbial populations that can survive and thrive in the conditions that exist at the site. As a result there is not any one mechanism, nor is there one bacteria that is responsible for the degradation

process. It is in fact a consortia of microorganisms and a variety of mechanisms that bring about the results.



**Figure 5** Hydrogenolysis.



**Figure 6** Cometabolic degradation.

PXY-1020, 343-44.

62. Fermentation of organic compounds to produce hydrogen ( $H_2$ ) has also been known, and can follow the reactions (PXY-1021, Rittmann, P.666):



63. Methods of dechlorination using zero valent metals and organic hydrogen donors in combination were also practiced in the United States 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 as described in the '709 Patent. PXY-1017, p. 12-13. 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, each for the same purpose as described in the '709 Patent. PXY-1005, p. 2-3.

64. In view of the state of the art as of January 6, 2003, the Challenged Claims were known before the priority date of the '709 Patent. 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. Dependent Claims 2-16

also represent no more than what was known and routinely practiced in the art, for example use of injection rods (Claim 3), colloidal suspensions (Claims 11, 15 and 16), and cleansing of lines after use (Claim 13).

**C. Prosecution History of the ‘709 Patent**

65. The ‘709 Patent issued on May 12, 2009, from U.S. App. No. 11/423,749 filed on June 13, 2006 (“the ‘749 Application”) 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”), with each claiming priority to U.S. Provisional Application Nos. 60/437,983 filed January 6, 2003 (“the ‘983 Application”).

66. A copy of the U.S. Patent and Trademark Office file wrapper for the ‘749 Application is marked as Exhibit PXY-1002. The only rejection issued by the examiner in the ‘749 Application was a nonstatutory double patenting rejection over Claims 1-10 of the Parent Patent.

**D. Priority Date of Claims**

67. As discussed in Section VI.C, the earliest priority claim for each of Claims 1-16 of the ‘709 Patent is to U.S. Provisional Patent App. No. 60/437,983 filed January 6, 2003 (the “‘983 Provisional Application”).

68. I have been informed and understand that for the purpose of this *inter partes* review, Petitioner does not challenge the claim of priority for the

Challenged Claims. I have therefore been instructed to assume that the effective filing date of each of the Challenged Claims is January 6, 2003.

## **VII. Claim Construction**

69. I have been informed and understand that in order to properly evaluate the ‘709 Patent, the terms of the claims must first be interpreted. I have also been informed and understand that during an *inter partes* review proceeding, the claims are to be given their broadest reasonable interpretation in light of the specification of the ‘709 Patent. I have further been informed and understand that claim terms are given their ordinary and accustomed meaning as would be understood by one of ordinary skill in the relevant art unless the inventor, as a lexicographer, has set forth a special meaning for a term.

70. In order to construe the claims, I have reviewed the entirety of the ‘709 Patent and its prosecution history. Consistent with these standards, I propose the below interpretation for certain claim terms.

71. I have been informed and understand that a rule of claim drafting known as “antecedent basis” states that claim elements are identified throughout a claim by designating the element with an indefinite article “a” on its first appearance in the claim and thereafter employing a definite article such as “the” or “said.” I have also been informed and understand that errors in claim drafting do not render a claim indefinite under 35 U.S.C. § 112 when the meaning of the claim

would be reasonably certain to a POSA when viewed in light of the claim language and specification.

**A. “organic hydrogen donor”**

72. This term appears in the ‘709 Patent’s only independent claim, Claim 1.

73. The term “*organic hydrogen donor*” is not a term of art and is not expressly defined in the specification. However, it is still used extensively in the ‘709 Patent to refer to several examples of organic hydrogen donors such as glucose, sucrose, sugars, alcohols, propionates, lactates, acetates, chitin, butyrate, poly lactate esters, glycerol tripoly lactate, xylitol pentapoly lactates, and sorbitol hexapoly lactate:

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.

organic hydrogen donor such as glucose, sucrose, alcohols, propionates, lactates, acetates, chitin, poly lactate esters, glycerol tripoly lactate, xylitol pentapoly lactate, and sorbitol hexapoly lactate 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 tripoly lactate, xylitol pentapoly lactate, or sorbitol hexapoly lactate, 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 tripoly lactate, xylitol pentapoly lactate, and sorbitol hexapoly lactate. PXY-1001, Claim 9

74. A POSA would understand the term “*organic hydrogen donor*” using the broadest reasonable interpretation to mean “a biodegradable organic compound capable of producing hydrogen through microbial activity.” This construction is consistent with the term’s use in the ‘709 Patent.

75. A POSA would further understand that the listed biodegradable organic compounds could be involved with more than one biotic reaction, including (1) anaerobic reductive dechlorination reactions where the amended organic functions as an electron donor, and Cl in the chlorinated organic contaminant molecules (e.g. trichloroethylene) are replaced by (donated) H, and (2) fermentative reactions with hydrogen gas (H<sub>2</sub>) as a possible product.

## **VIII. Challenge #1: Claims 1, 7, 9, and 14 are anticipated by Hitchens**

### **A. Hitchens Teaches Every Element of Claims 1, 7, 9, and 14**

76. Hitchens describes degrading chlorinated solvents with granular iron (zero valent iron) and introducing glucose, lactate or formates (organic hydrogen donors) into the soil. 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 contaminates.” PXY-1003, 3:3:39-56.

77. 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 an organic hydrogen donor. PXY-1003, 1:10-15. Claims 42-44 of Hitchens disclose a method corresponding to the claimed 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;  
dissolving at least one organic electron donor in water, wherein the at least one organic electron donor is selected from glucose, methanol, ethanol, toluene, dichloromethane, an acid, a salt of the acid and mixtures thereof, wherein the acid is selected from acetic acid,

formic acid, lactic acid, benzoic acid, propionic acid, crotonic acid, and butyric acid; and delivering the water into the environment by electroosmosis.

43. The method of claim 42, wherein the step of generating hydrogen includes corroding metal particles dispersed in the soil.

44. The method of claim 42, further comprising the step of electrokinetically delivering nutrients into the soil.

PXY-1003, Claims 42-44.

78. Hitchens discloses a method for soil and ground water remediation utilizing both biotic and abiotic mechanisms.

79. Regarding the biotic mechanism, Hitchens discloses the advantages of “supplying hydrogen ( $H_2$ ) 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.

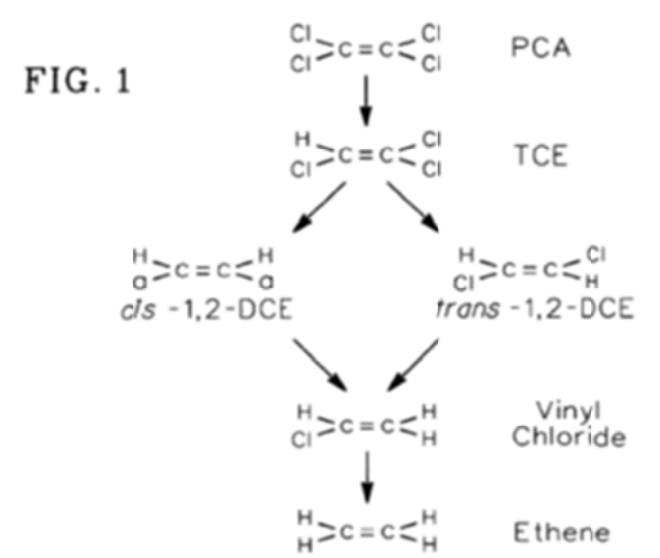
80. Hitchens describes that chlorinated solvents can be degraded in abiotic reactions with granular iron. The reactions include:  $Fe^0 \rightarrow Fe^{2+} + 2e^-$ ;  $C_2HCl_3 + 3H^+ + 6e^- \rightarrow C_2H_4 + 3Cl^-$  and the subsequent formation of hydrogen gas  $2H_2O + 2e^- \rightarrow H_2(g) + 2OH^-$ . PXY-1003, 3:39-56.

81. 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.” PXY-1003, 3:57-60. Hitchens further identifies that the availability of a suitable electron donor (organic hydrogen donor) for this process is critical:

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

82. Fig 1. Illustrates this mechanism:



83. Hitchens discloses “vitamin B<sub>12</sub> (Co-containing porphyrin) which is often found in bacteria, and coenzyme F<sub>430</sub> (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.

84. 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. Hitchens states:

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.

## **B. Detailed Invalidity Analysis**

85. The following table explains how Hitchens teaches every element of Claims 1, 7, 9, and 14 of the ‘709 Patent, alone or in combination with the knowledge, skill, and creativity of a POSA.

<b>‘709 Patent Claims</b>	<b>Disclosure in Hitchens (PXY-1003)</b>
[1.0] A method for accelerated anaerobic dechlorination of subsoil matter, comprising the steps of:	Hitchens discloses this claim element.  Hitchens describes that the toxicity of many contaminants can be decreased or eliminated by reductive dechlorination, which involves successive

'709 Patent Claims	Disclosure in Hitchens (PXY-1003)
	<p>shedding of chlorine atoms under reduced anaerobic condition that is biologically catalyzed. PXY-1003, 1:18-25.</p> <p>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.</p> <p>Hitchens discloses a method for accelerated anaerobic dechlorination of subsoil matter.</p>
<p>[1.1] supplying a mixture including a zero valent metal into soil pathways to biologically react with the dissolved chlorinated solvents in the groundwater; and</p>	<p>Hitchens discloses this claim element.</p> <p>Hitchens discloses “[c]hlorinated solvents, such as trichloroethylene and perchloroethylene can be degraded by reactions with <b>granular iron</b>. This abiotic reductive dehalogenation is thought to proceed with pseudo-first order kinetics” PXY-1003, 3:39-56.</p> <p>Hitchens discloses the governing equations for the reductive dehalogenation (dechlorination) includes a mixture of zero valent iron and magnesium:</p> $(1) \text{Fe}^{\circ} \rightarrow \text{Fe}^{2+} + 2e^{-}$ $(2) \text{C}_2\text{HCl}_3 + 3\text{H}^{+} + 6e^{-} \rightarrow \text{C}_2\text{H}_4 + 3\text{Cl}^{-}$ <p>PXY-1003, 3:49-51.</p> <p>Hitchens further discloses “TCE [<math>\text{C}_2\text{HCl}_3</math>] degrades spontaneously in the presence of both water and iron, requiring no additives or application of energy” PXY-1003, 4:1-3. In Hitchens, the “iron is placed deep</p>

'709 Patent Claims	Disclosure in Hitchens (PXY-1003)
	<p>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.</p> <div data-bbox="673 478 1365 1024" data-label="Image"> </div> <p>Hitchens also describes how particles used in the invention may be distributed in the soil, by placing them in a trench, injection them into holes or wells or packages into porous filter socks. PXY-1003, 12:18-29.</p> <p>The zero valent iron in the mixture is involved in a multitude of reactions, including reactions with the dissolved chlorinated solvents in the groundwater. The addition of zero valent iron to the subsurface that is under reducing conditions (anaerobic) will result in the formation of <math>H_2</math> gas as disclosed by Hitchens.</p> <p>“In the ‘managed’ production of hydrogen, water-induced corrosion leading to an inactive iron hydroxide surface (e.g., eq. 8) is avoided because the iron surface is corrosion protected by the magnesium, which functions as a sacrificial anode.</p>

'709 Patent Claims	Disclosure in Hitchens (PXY-1003)
	<p> <math>\text{Fe}^{2+} + 2\text{H}_2\text{O} \rightarrow \text{Fe}(\text{OH})_2 + 2\text{H}^+</math> (8)  <math>\text{Fe} + 2\text{H}^+ \rightarrow \text{Fe}^{2+} + \text{H}_2</math> (9)" PXY-1003, 11:33-41. </p> <p> This hydrogen gas produced enhances the anaerobic biodegradation process of the dissolved chlorinated solvents in the soil by indigenous anaerobic bacteria. "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. </p>
<p>[1.2] supplying an organic hydrogen donor into the soil pathways to produce dechlorinating conditions such that indigenous anaerobic bacteria biodegrade residual concentrations of chlorinated solvents.</p>	<p>Hitchens discloses this claim element.</p> <p>Hitchens discloses "supplying hydrogen (<math>\text{H}_2</math>) 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.</p> <p>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.</p> <p>As described by Hitchens, supplying an organic</p>



‘709 Patent Claims	Disclosure in Hitchens (PXY-1003)
	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.
[7.0] the method of claim 1 wherein the mixture further includes nutrients.	<p>Hitchens discloses this claim element.</p> <p>Hitchens discloses the step of “delivering nutrients into the soil” in addition to the elemental iron. PXY-1003, 16:33-34.</p>
[9.0] 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.	<p>Hitchens discloses this claim element.</p> <p>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 <i>lactate</i>) were used.” PXY-1003, 2:32-44 (emphasis added).</p>
[14.0] the method of claim 1 wherein said metal is iron.	<p>Hitchens discloses this claim element.</p> <p>As discussed above, “[c]hlorinated solvents, such as trichloroethylene and perchloroethylene can be degraded by reactions with granular iron.” PXY-1003, 3:39-56.</p> <p>(1) <math>\text{Fe}^{\circ} \rightarrow \text{Fe}^{2+} + 2e^{-}</math></p> <p>(2) <math>\text{C}_2\text{HCl}_3 + 3\text{H}^{+} + 6e^{-} \rightarrow \text{C}_2\text{H}_4 + 3\text{Cl}^{-}</math></p>

'709 Patent Claims	Disclosure in Hitchens (PXY-1003)
	<p>PXY-1003, 3:49-51.</p> <p>Hitchens discloses the zero valent metal used for reductive dechlorination is iron (<math>\text{Fe}^0</math>).</p>

**IX. Challenge #2: Claims 1, 3, 7, 9, and 14-16 are anticipated by Vance**

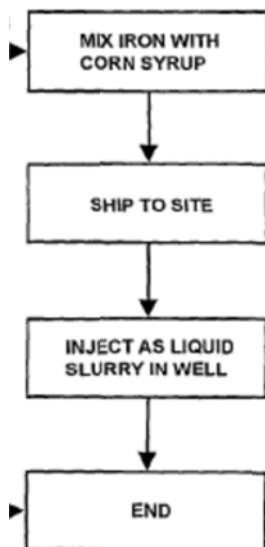
**A. Vance Teaches Every Element of Claims 1, 3, 7, 9, and 14-16**

86. 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. 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. Corn syrup contains sugars, and the '709 Patent expressly identifies "sugars" as an organic hydrogen donor. PXY-1001, 4:29-32, 6:57-60.

87. Vance explains that "since the mid-1990, there have been a series of dramatic developments for the in-situ treatment of chlorinated solvents," and describes a method "based on the sequential reduction of chlorinated hydrocarbons to innocuous end products." PXY-1010, ¶ [0002]. 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.

PXY-1010, ¶ [0121], Fig. 5.



PXY-1010, Fig. 5 (truncated).

88. Some methods disclosed in Vance are directed to “using elemental metal to reductively dehalogenate halogenated hydrocarbons.” *Id.* at Abstract.

89. Vance clearly discloses the use of zero valent metals in dehalogenation:

When elemental iron is in the presence of chlorinated compounds (contaminants) a naturally occurring reaction takes place and the chlorinated compounds are reduced and form harmless compounds. The production of extremely small metal particles containing elemental iron or a mixture of elemental iron and a second metal is imperative to this method of treating contaminants and is the subject of the present invention. *Id.* at Abstract.

The process exploits the use of zero valence state elemental metals to reductively dehalogenate halogenated hydrocarbons. *Id.* at ¶ [0002].

The most common metal being utilized for this purpose is iron. But other metals including tin, zinc, and palladium have also shown to be effective. The process may be best described as anaerobic corrosion of the metal by the chlorinated hydrocarbon. During this process, the hydrocarbon is adsorbed directly to the metal surface where the dehalogenation reactions occur. Increasing surface area (by reducing the size of iron particles) increases the effectiveness of the process. *Id.* at ¶ [0003].

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. *Id.* at ¶ [0112].

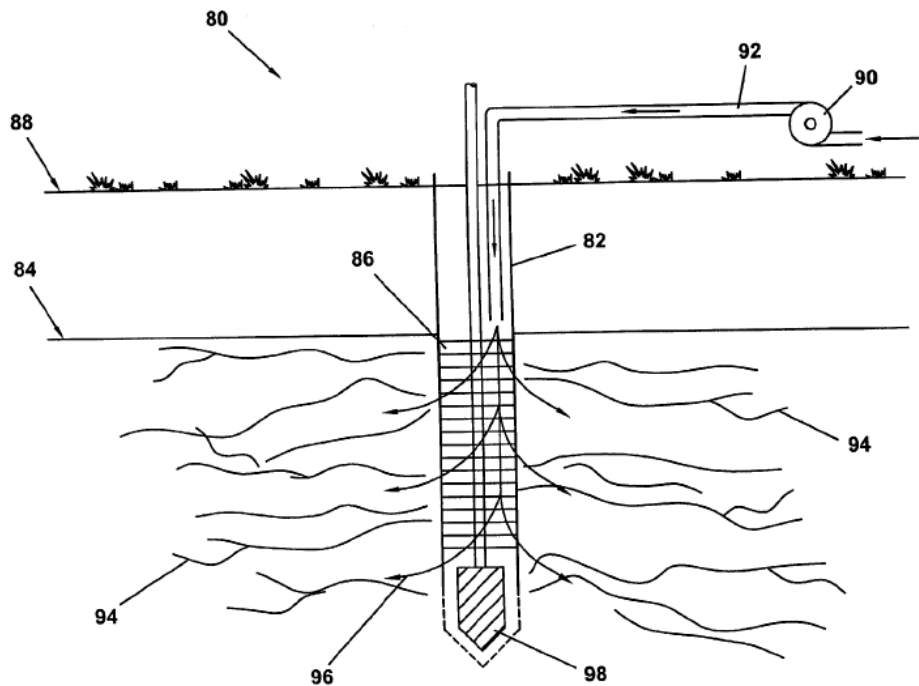
90. Vance discloses three mechanisms by which elemental iron reduce halogenated compounds: as a direct reductant, as a catalyst for a hydrogen reaction, and as an indirect or solubilized reductant. *Id.* at ¶¶ [0004] – [0007].

91. The methods of Vance are intended to be used in an anaerobic environment. Vance notes that “[t]he elemental metal may be kept in an elemental state by keeping it in an oxygen-scavenging environment” and that the disclosed method “may be best described as anaerobic corrosion of the metal by the chlorinated hydrocarbon.” *Id.*, Abstract, ¶ [0003].

92. The zero valent metal used in Vance is injected into subsoil and groundwater:

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 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. *Id.* at ¶ [0104].



**FIG. 4**

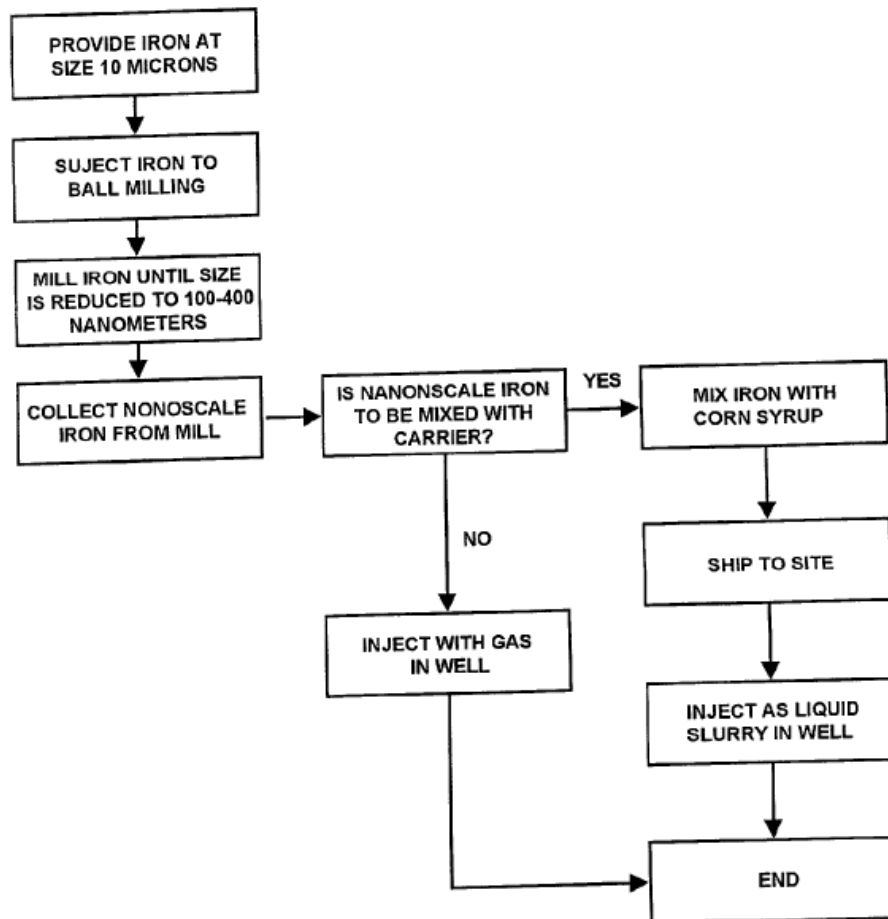
PXY-1010, Fig. 4.

93. Vance further describes suspending the particulate zero valent metal in a carbohydrate such as corn syrup:

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. *Id.* at ¶¶ [0118] – [0120].



**FIG.5**

PXY-1010, Fig. 5.

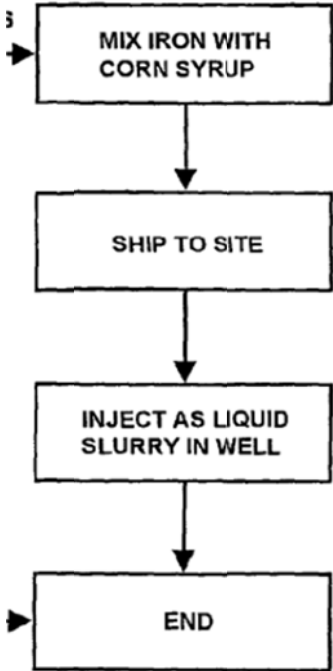
## B. Detailed Invalidity Analysis

94. The following table explains how Vance teaches every element of Claims 1, 3, 7, 9, and 14-16 of the '709 Patent, alone or in combination with the knowledge, skill, and creativity of a POSA.

'709 Patent Claims	Disclosure in Vance (PXY-1010)
[1.0] A method for accelerated anaerobic dechlorination of subsoil matter, comprising the steps of:	<p>Vance discloses this claim element.</p> <p>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]. The process utilized by Vance “may be best described as anaerobic corrosion of the metal by the chlorinated hydrocarbon.” PXY-1010, ¶ [0003].</p>
[1.1] supplying a mixture including a zero valent metal into soil pathways to biologically react with the dissolved chlorinated solvents in the groundwater; and	<p>Vance discloses this claim element.</p> <p>Vance describes injecting/supplying a colloid suspension having elemental metal “mixed with a second metal”, a carbohydrate, and 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.</p> <p>As would be understood by a POSA, 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, ¶ [0112].</p>

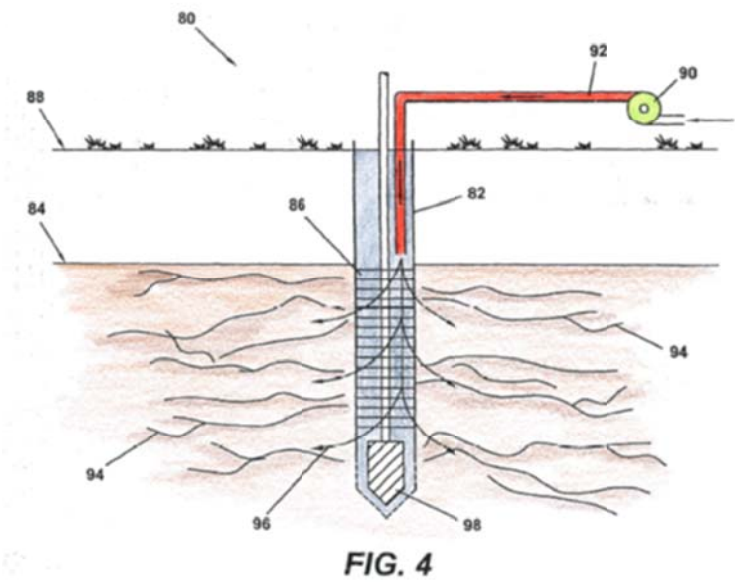
‘709 Patent Claims	Disclosure in Vance (PXY-1010)
	<p>“Additionally, the present invention is to a method of injecting nanoscale metal particles into soil, comprising the steps of: a. making a colloid suspension having metal particles in the presence of a carbohydrate; and b. injecting said colloid suspension into the soil through a well at a flow rate sufficient to move the colloid suspension through the soil.” PXY-1010, ¶¶ [0118-0120].</p> <p>“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].</p> <p>Vance in Fig. 5 (below) provides an example of supplying a mixture including elemental iron into the soil:</p>



'709 Patent Claims	Disclosure in Vance (PXY-1010)
	 <pre> graph TD     A[MIX IRON WITH CORN SYRUP] --&gt; B[SHIP TO SITE]     B --&gt; C[INJECT AS LIQUID SLURRY IN WELL]     C --&gt; D[END] </pre> <p>PXY-1010, Fig. 5 (truncated).</p> <p>The zero valent iron in the mixture is involved in a multitude of reactions, including reactions with the dissolved chlorinated solvents in the groundwater.</p> <p>The addition of zero valent iron the subsurface that is under reducing conditions (anaerobic conditions as described by Vance) will result in the formation of H<sub>2</sub> gas. 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.</p>
<p>[1.2] supplying an organic hydrogen donor into the soil pathways to produce dechlorinating conditions such that indigenous anaerobic bacteria biodegrade</p>	<p>Vance discloses this claim element.</p> <p>Vance describes supplying a colloidal suspension of an organic hydrogen donor, specifically corn syrup, into the soil pathways. PXY-1010, ¶¶ [0118]-[0122], Fig. 5, Claims 11, 15, and 18.</p>

'709 Patent Claims	Disclosure in Vance (PXY-1010)
residual concentrations of chlorinated solvents.	<div data-bbox="831 327 1162 997" data-label="Diagram"> <pre> graph TD     A[MIX IRON WITH CORN SYRUP] --&gt; B[SHIP TO SITE]     B --&gt; C[INJECT AS LIQUID SLURRY IN WELL]     C --&gt; D[END] </pre> </div> <p data-bbox="609 1081 1421 1417">“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].</p> <p data-bbox="609 1501 1437 1837">“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].</p>

‘709 Patent Claims	Disclosure in Vance (PXY-1010)
	<p>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.</p>
<p>[3.0] 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.</p>	<p>Vance discloses this claim element.</p> <p>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.</p> <p>“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, ¶ [0121].</p>

'709 Patent Claims	Disclosure in Vance (PXY-1010)
	<div data-bbox="609 289 1339 871">  </div> <p data-bbox="609 955 1446 1386"> “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 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]. </p> <p data-bbox="609 1459 1446 1795"> “FIG. 2 illustrates another method of the present invention 30 for injecting the nanoscale metal 46 of the present invention by pump 40 through an injector 42 into a open bore hole 32 through a screen 36 into a plume 44 that is located beneath ground level 38 and beneath the water table 34. Non-contaminated water 48 beneath the water table is also illustrated.” PXY-1010, ¶ [0102]. </p>

‘709 Patent Claims	Disclosure in Vance (PXY-1010)
<p>[7.0] the method of claim 1 wherein the mixture further includes nutrients.</p>	<p>Vance discloses this claim element.</p> <p>Vance further describes supplying, along with the zero valent iron in a colloidal suspension, a carbohydrate atomized in nitrogen: “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.</p> <p>As described in the ‘709 Patent, nitrogen is a nutrient: “A solution of sodium sulfite and nutrients (nitrogen and ortho-phosphate) is immediately injected into the subsurface pathways and voids that were developed during the gas injection step.” PXY-1001, 2:64-67.</p>
<p>[9.0] 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 pentapoly lactate, and sorbitol hexapoly lactate.</p>	<p>Vance discloses this claim element.</p> <p>Vance describes supplying a colloidal suspension of an organic hydrogen donor, specifically corn syrup, into the soil pathways. PXY-1010, ¶¶ [0118-0120], [0122], Fig. 5.</p> <p>Corn syrup contains sugars.</p>
<p>[14.0] the method of claim 1 wherein said metal is iron.</p>	<p>Vance discloses this claim element.</p> <p>Vance discloses the metal is iron. <i>See</i> [1.1] (above).</p> <p>Vance claims the use of elemental iron (zero valent metal): “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.”</p>

‘709 Patent Claims	Disclosure in Vance (PXY-1010)
	PXY-1010, Claim 2.
[15.0] The method of claim 1 wherein said metal is in a colloidal suspension.	<p>Vance discloses this claim element.</p> <p>Vance discloses a colloidal suspension of iron powder and corn syrup:</p> <p>“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], Claim 18.</p> <p><i>See also</i> PXY-1010, Fig. 5.</p>
[16.0] The method of claim 15 wherein the colloidal suspension includes a reducing agent.	<p>Vance discloses this claim element.</p> <p>Vance describes supplying a colloidal suspension of an organic hydrogen donor, specifically corn syrup, into the soil pathways. PXY-1010, ¶¶ [0118-0120], [0122], Fig. 5, Claim 18.</p> <p>Corn syrup is a known reducing agent.</p>

**X. Challenge #3: Claims 1, 3, 7, and 14 are anticipated by Alvarez**

**A. Alvarez Teaches Every Element of Claims 1, 3, 7, and 14**

95. 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. The method of Alvarez is a “reductive treatment using zero valent iron and anaerobic biotransformations.” PXY-1004, Abstract.

96. Alvarez describes reductive dechlorination by iron corrosion, which produces H<sub>2</sub> and, as the dechlorination method is anaerobic, also “support[s] the growth of useful bacteria.” PXY-1004, p. 7. 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. 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.

97. 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.

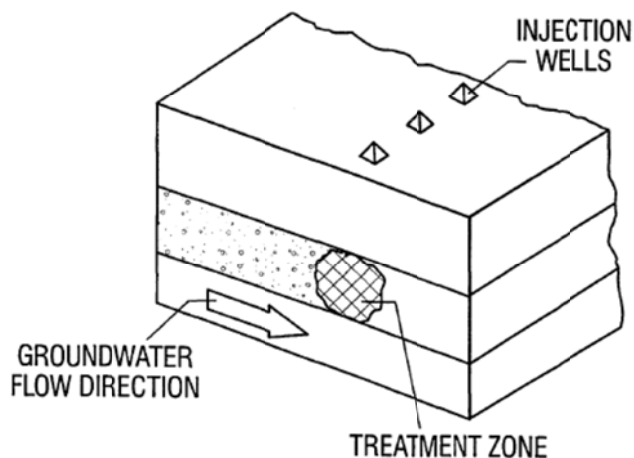


FIG. 2

PXY-1004, Fig 2.

98. Alvarez describes *in situ* remediation may be achieved using a combination/mixture including iron 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.

99. 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. 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 is an electron donor such as methanol or acetate. PXY-1004, pp. 23-24.



100. Alvarez discloses injecting zero valent iron and an organic hydrogen donor into the soil pathways for soil dehalogenation as described in Claims 1, 3, 7 and 14.

## **B. Detailed Invalidity Analysis**

101. The following table explains how Alvarez teaches every element of Claims 1, 3, 7, and 14 of the ‘709 Patent, alone or in combination with the knowledge, skill, and creativity of a POSA.

<b>‘709 Patent Claims</b>	<b>Disclosure in Alvarez (PXY-1004)</b>
[1.0] A method for accelerated anaerobic dechlorination of subsoil matter, comprising the steps of:	<p>Alvarez discloses this claim element.</p> <p>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.</p> <p>Alvarez discloses the method results in reductive dechlorination: “[p]olychlorinated organics can also be reduced using the e- 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.</p> <p>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.</p>
[1.1] supplying a mixture including a zero valent metal into soil pathways	Alvarez discloses this claim element.

‘709 Patent Claims	Disclosure in Alvarez (PXY-1004)
<p>to biologically react with the dissolved chlorinated solvents in the groundwater; and</p>	<p>Alvarez’s dechlorination method supplies a composition / mixture containing zero-valent iron and bacteria culture to make contact with a contaminate plume in the ground:</p> <p>“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. <i>See also</i> PXY-1004, pp. 6-11, Claim 35.</p> <p>The composition (mixture) of Alvarez includes zero valent iron:</p> <p>“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.</p> <p>“tetrachloroethene (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</p>

‘709 Patent Claims	Disclosure in Alvarez (PXY-1004)
	<p>the H<sub>2</sub> from Fe(O) corrosion and support the growth of useful anaerobic bacteria.” PXY-1004, p. 7-8.</p> <p>“the 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</p> <p>“In a first embodiment, the invention concerns a device which comprises a composition of zero-valent iron and a culture of one or more hydrogenotrophic bacteria.” PXY-1004, p. 9.</p> <p>The zero valent iron in the composition/mixture is involved in a multitude of reactions, including reactions with the dissolved chlorinated solvents in the groundwater.</p> <p>The addition of zero valent iron 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. This hydrogen gas enhances the anaerobic biological degradation process of the residual dissolved chlorinated solvents by the indigenous anaerobic bacteria. In addition, “[t]he dechlorination method is anaerobic and the indigenous anaerobic bacteria in the soil biologically degrade the dissolved chlorinated solvents. The mixture/composition as described in Alvarez also contains a culture of hydrogenotrophic bacteria to remove target contaminants and likewise biologically reacts with chlorinated solvents in the soil.</p>

'709 Patent Claims	Disclosure in Alvarez (PXY-1004)
<p>[1.2] supplying an organic hydrogen donor into the soil pathways to produce dechlorinating conditions such that indigenous anaerobic bacteria biodegrade residual concentrations of chlorinated solvents.</p>	<p>Alvarez discloses this claim element.</p> <p>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.</p> <p>Alvarez notes “organic substrates that are commonly used to support biological denitrification” are methanol and acetate. PXY-1004, p. 26. 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; Freedman and Gossett, 1989; Fathepure and Boyd, 1988).” PXY-1004 p. 58.</p> <p>Methanol and acetate are described as electron donors by Alvarez. PXY-1004 pp. 23-24.</p> <p>“Electron donors that have been investigated for methanogenic biotransformation of [chlorinated aliphatic hydrocarbons] include <i>methanol</i> (Mikesell and Boyd, 1990; Bagley and Gossett, 1995), <i>acetate</i> (Bouwer et al., 1981; Bouwer and McCarty, 1983; Hughes and Parkin, 1996), and hydrogen (DiStefano et al., 1992).” PXY-1004 pp. 23-24 (emphasis added).</p> <p>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.</p>

<b>'709 Patent Claims</b>	<b>Disclosure in Alvarez (PXY-1004)</b>
<p>[3.0] 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.</p>	<p>Alvarez discloses this claim element.</p> <p>Alvarez describes the injection of the composition into the ground using an injection system.</p> <p>“the 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.</p> <p>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.</p>
<p>[7.0] the method of claim 1 wherein the mixture further includes nutrients.</p>	<p>Alvarez discloses this claim element.</p> <p>Alvarez describes the addition of nutrients in the composition / mixture with zero valent metal. PXY-1004 p. 11.</p> <p>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.</p>

'709 Patent Claims	Disclosure in Alvarez (PXY-1004)
[14.0] the method of claim 1 wherein said metal is iron.	<p>Alvarez discloses this claim element.</p> <p>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.</p>

**XI. Challenge #4: Claims 1-3, 5, 7, 11, 12, and 14-16 are obvious over Rice in view of Cantrell**

**A. Rice in View of Cantrell Teaches Every Element of Claims 1-3, 5, 7, 11, 12, and 14-16**

102. Rice in view of Cantrell discloses the claimed subject matter of the Challenged Claims.

103. 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.

104. Rice is focused on the decontamination of groundwater at sites having “[h]alogenated organic compounds, particularly aliphatic organics such as the chloroethanes and chloroethenes, especially tetrachloroethene (also known as perchloroethene or PCE), trichloroethene (TCE), dichloroethene (DCE) and vinyl chloride (VC).” *Id.*, 1:13-17.

105. Rice discloses a method for decontaminating such compounds using indigenous anaerobic microorganisms:

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. *Id.*, 4:29-36.

106. Rice supplies an energy source and nutrients – through groundwater injection – to these indigenous anaerobic microorganisms to stimulate reductive dehalogenation:

A suitable electron donor substrate is an organic molecule that can be utilized as an energy source by the indigenous anaerobic microorganisms. 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. A most preferred electron donor is lactate which is relatively inexpensive and has a high hydrogen:carbon ratio. If lactate is used as the electron donor, it can be provided in the form of sodium lactate, which can be adjusted as needed to a neutral pH. One skilled in the art can select other electron donors which can include sugars, organic acids, saccharides, polysaccharides, other long-chain branched or unbranched organic compounds, and the like.

The aqueous solution can optionally contain trace nutrients for enhancing anaerobic biodegradation. A suitable trace nutrient is yeast extract which is commercially available from, for example, Fisher

Scientific. The trace nutrients can be added to a concentration of 2 mg/l in the injected solution, although the exact amount is not critical. *Id.*, 4:7-28.

The method includes the step of injecting in situ, into a groundwater-saturated matrix upgradient of, or within an area that has been impacted by, a halogenated organic compound, a deoxygenated aqueous solution that comprises an electron donor. The deoxygenated solution is injected in an amount sufficient to establish an anaerobic treatment zone that comprises the anaerobic microorganism. The amount of dissolved oxygen in the deoxygenated solution is sufficiently low that the solution cannot support growth of aerobic microorganisms in the treatment zone, typically about 1 mg/L or less. The electron donor is injected in an amount sufficient to support reductive dehalogenation of the organic compound in the treatment zone by the anaerobic microorganism. *Id.*, 2:21-33.

107. Cantrell discloses a method of making an in-situ chemical barrier:

A chemical barrier is formed by injecting a suspension of solid particles or colloids into the subsurface. First, a stable colloid suspension is made including a surfactant and a non-Newtonian fluid. ... A second step involves injecting the optimized stable colloid suspension at a sufficiently high flow rate to move the colloids through the subsurface sediment. PXY-1026, Abstract.

108. The barrier described by Cantrell is “a zone in subsurface environment that is capable of intercepting chemicals and preventing passage of the chemicals beyond the zone.” PXY-1026, 3:38-41. The barrier is used for the treatment of soil contaminated with “chemical liquids” such as halogenated hydrocarbon compounds. PXY-1026, 1:20, 1:31-32.



109. Cantrell teaches the injection of iron ( $\text{Fe}^0$ ) as a colloidal suspension into the subsurface to remediate contaminants:

A chemical barrier is formed by injecting a suspension of solid particles or colloids into the subsurface. First, a stable colloid suspension is made. This stable colloid suspension is characterized by colloid concentration, colloid size, colloid material, solution ionic strength, and chemical composition. A second step involves injecting the optimized stable colloid suspension at a sufficiently high flow rate to move the colloids through the subsurface sediment, but not at such a high rate so as to induce resuspension of indigenous soil particles in the aquifer. PXY-1026, 2:11-19.

The selection of the materials used in a chemical barrier or zone is dependent on the targeted contaminant, the remediation objective, and site conditions (such as competing ions, sediment texture, and water flow rate) Preferred materials are good sequestering agents, inexpensive and possess a sufficiently high hydraulic conductivity to promote groundwater flow through the chemical barrier. Materials useful according to the present invention include zeolite minerals, minerals modified with surface-bound chelates and quaternary amines, organic peat, limestone, titanium hydroxide, titanium oxide, fly ash, saw dust, lignite, hematite, magnetite, goethite, hydroxyapatite, andisols for anions, and zero-valent iron,  $\text{Fe}^0$ . Solid particle sizes of any or a combination of the above recited materials are preferably less than about 5 micron. *Id.* at 5:17-32.

110. Cantrell teaches a preference for zero-valent iron in decontamination efforts, particularly those involving halogenated contaminants:

Zero-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, 5:32-35.

**B. Motivation to Combine Rice and Cantrell**

111. 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. 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.

112. 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.

113. 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. The proposed combination would result in the claimed invention of the Challenged Claims.

### **C. Detailed Invalidity Analysis**

114. The following table explains how Rice in view of Batchelor teaches every element of Claims 1-3, 5, 7, 11, 12, and 14-16 of the '709 Patent, alone or in combination with the knowledge, skill, and creativity of a POSA.

<b>'709 Patent Claims</b>	<b>Disclosure in Rice in view of Cantrell</b>
[1.0] A method for accelerated anaerobic dechlorination of subsoil matter, comprising the steps of:	<p>Rice in view of Cantrell discloses this claim element.</p> <p>Rice discloses in situ anaerobic dechlorination of a ground water plume:</p> <p>“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.</p>

‘709 Patent Claims	Disclosure in Rice in view of Cantrell
<p>[1.1] supplying a mixture including a zero valent metal into soil pathways to biologically react with the dissolved chlorinated solvents in the groundwater; and</p>	<p>Rice in view of Cantrell discloses this claim element.</p> <p>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. However, the mixture does not include zero valent metal:</p> <p>“A suitable electron donor substrate is an organic molecule that can be utilized as an energy source by the indigenous anaerobic microorganisms. The electron donor substrate should be a relatively simple organic molecule having available hydrogens that can act as the proximate electron donor during dehalogenation.” PXY-1006, 4:7-12.</p> <p>“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, Dehalospirillum, and Dehalobacter (in particular, Desulfomonile tiedjei, Dehalospirillum 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.” <i>Id.</i>, 4:29-43.</p> <p>Cantrell discloses a method to dechlorinate dissolved chlorinated solvents in ground water which includes the introduction of zero valent metal into the soil pathways.</p>

‘709 Patent Claims	Disclosure in Rice in view of Cantrell
	<p>“A chemical barrier is formed by injecting a suspension of solid particles or colloids into the subsurface. First, a stable colloid suspension is made. This stable colloid suspension is characterized by colloid concentration, colloid size, colloid material, solution ionic strength, and chemical composition. A second step involves injecting the optimized stable colloid suspension at a sufficiently high flow rate to move the colloids through the subsurface sediment, but not at such a high rate so as to induce resuspension of indigenous soil particles in the aquifer.” PXY-1026, 2:11-19.</p> <p>“The selection of the materials used in a chemical barrier or zone is dependent on the targeted contaminant, the remediation objective, and site conditions (such as competing ions, sediment texture, and water flow rate) Preferred materials are good sequestering agents, inexpensive and possess a sufficiently high hydraulic conductivity to promote groundwater flow through the chemical barrier. Materials useful according to the present invention include zeolite minerals, minerals modified with surface-bound chelates and quaternary amines, organic peat, limestone, titanium hydroxide, titanium oxide, fly ash, saw dust, lignite, hematite, magnetite, goethite, hydroxyapatite, andisols for anions, and zero-valent iron, Fe<sup>0</sup>. Solid particle sizes of any or a combination of the above recited materials are preferably less than about 5 micron.” <i>Id.</i> at 5:17-35.</p> <p>“A method of injecting solid particles into subsurface soil, comprising the steps of: (a) making a colloid suspension having said solid particles in water; (b) adding a surfactant to said suspension and further adding a non-Newtonian fluid thereby making a non-Newtonian stable colloid suspension; and (b) injecting the stable colloid suspension into the subsurface soil through a well at a flow rate sufficient to move the stable colloid suspension through the subsurface</p>

‘709 Patent Claims	Disclosure in Rice in view of Cantrell
	<p>sediment and without substantially resuspending indigenous soil particles in the subsurface soil.” PXY-1026, Claim 1.</p> <p>“The method as recited in claim 1, wherein said solid particles are <math>\text{Fe}^0</math>.” PXY-1026, Claim 13.</p> <p>Cantrell additionally describes “[w]hen zero-valent iron metal particles are used as the solid particles, suspensions of <math>\text{Fe}^0</math> colloids (<math>2\pm 1\ \mu\text{m}</math>) are injected at a colloid injection rate, <math>P\leq 0.01</math>, and concentration, <math>P\sim 0.05</math>, to uniformly distribute colloids throughout soil columns at concentrations <math>&gt;0.3\%</math> (w/w).” PXY-1026, 5:55-60.</p> <p>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.</p> <p>Cantrell further describes many experiments utilizing zero valent colloids:</p> <p>“An experiment was conducted to evaluate the ability of a commercially available <math>\text{Fe}^0</math> colloid (Product Number S3700; International Specialty Products, Wayne, N.J.) to remove an oxidized dissolved species from solution.” PXY-1026, 6:50-54.</p> <p>“Suspensions of <math>\text{Fe}^0</math> colloids were introduced into sand columns at several flow rates and influent colloid concentration.” PXY-1026, 8:59-61.</p> <p>“An experiment was conducted to demonstrate three shearthinning fluids with <math>\text{Fe}^0</math> as colloids for emplacement in columns containing coarse-grained</p>

‘709 Patent Claims	Disclosure in Rice in view of Cantrell
	<p>sand.” PXY-1026, 10:57- 59.</p> <p>“To test the effectiveness of the VP for enhancing the emplacement of <math>\text{Fe}^0</math> colloids in porous media for greater distances, a 3-m long column experiment was conducted. In this case, the suspension of 1.0% <math>\text{Fe}^0</math> colloids in 0.01% VP was injected at a darcy velocity of <math>0.154 \text{ cm sec}^{-1}</math> and a three pore volume throughput (FIG. 7).” PXY-1026, 13:62-67.</p>
<p>[1.2] supplying an organic hydrogen donor into the soil pathways to produce dechlorinating conditions such that indigenous anaerobic bacteria biodegrade residual concentrations of chlorinated solvents.</p>	<p>Rice in view of Cantrell discloses this claim element.</p> <p>Rice discloses supplying organic hydrogen donors such as lactate, propionate and acetate into the soil:</p> <p>“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.</p> <p>“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: 9-13. <i>See also</i> PXY-1006, Claim 1 and 6.</p> <p>The zero valent iron in the mixture undergoes many reactions in the subsurface, including reactions with the dissolved chlorinated solvents in the groundwater. The addition of zero valent iron the subsurface that is under reducing conditions (anaerobic) will result in the formation of <math>\text{H}_2</math> gas as known in the art. This hydrogen gas enhances the anaerobic biological degradation process of the residual dissolved</p>

‘709 Patent Claims	Disclosure in Rice in view of Cantrell
	<p>chlorinated solvents by the indigenous anaerobic bacteria. The mixture also contains suitable anaerobic microorganisms to remove target contaminants and likewise promotes biological reactions involving chlorinated solvents in the soil. As described by Rice and as known in the art, 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.</p>
<p>[2.0] 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.</p>	<p>Rice in view of Cantrell discloses this claim element.</p> <p>Rice describes the addition of a reducing agent/deoxygenating agent, specifically sodium sulfite into the aqueous solution to deoxygenate water.</p> <p>“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, 3:47-57. <i>See also</i> Claims 1, 3, and 4.</p> <p>The aqueous solution is injected into soil pathways.</p> <p>“The method includes the step of injecting in situ into a groundwater-saturated matrix within or upgradient of a source of the organic compound a deoxygenated aqueous solution that comprises an electron donor to facilitate reductive dehalogenation of the organic compound.”PXY-1006, Abstract.</p> <p>“1. A method for dehalogenating an halogenated organic compound in groundwater, the method comprising the step of:</p>



‘709 Patent Claims	Disclosure in Rice in view of Cantrell
	<p>injecting into a saturated matrix within or upgradient of a source of the organic compound a deoxygenated aqueous solution that consists essentially of an electron donor ...” PXY-1006, Claim 1.</p> <p><i>See also</i> PXY-1006, 4: 9-10.</p>
<p>[3.0] 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.</p>	<p>Rice in view of Cantrell discloses this claim element.</p> <p>Rice describes the injection of the solution into the ground under pressure.</p> <p>“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</p> <p>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.</p>
<p>[5.0] The method of claim 2 wherein said reducing agent is sodium sulfite.</p>	<p>Rice in view of Cantrell discloses this claim element.</p> <p>Rice describes the addition of a reducing agent/deoxygenating agent, specifically sodium sulfite</p>

‘709 Patent Claims	Disclosure in Rice in view of Cantrell
	<p>into the aqueous solution to deoxygenate water.</p> <p>“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, 3:47-57. <i>See also</i> Claims 1, 3, and 4.</p>
<p>[7.0] the method of claim 1 wherein the mixture further includes nutrients.</p>	<p>Rice in view of Cantrell discloses this claim element.</p> <p>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.</p> <p>“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, Dehalospirillum, and Dehalobacter (in particular, Desulfomonile tiedjei, Dehalospirillum 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.</p>

‘709 Patent Claims	Disclosure in Rice in view of Cantrell
	<p>“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.</p> <p>“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.</p>
<p>[11.0] The method of claim 1 wherein said mixture including a zero valent metal is a colloidal suspension in a sodium sulfite solution.</p>	<p>Rice in view of Cantrell discloses this claim element.</p> <p>Cantrell discloses a method to dechlorinate dissolved chlorinated solvents in ground water which includes the introduction of zero valent metal in a colloidal suspension into the soil pathways. PXY-1026, 2:11-19; 5:17-35, Claims 1, 13.</p> <p>Specifically Cantrell describes “[w]hen zero-valent iron metal particles are used as the solid particles, suspensions of Fe<sup>0</sup> colloids (2±1 μm) are injected at a colloid injection rate, P≤0.01, and concentration, P~0.05, to uniformly distribute colloids throughout soil columns at concentrations &gt;0.3% (w/w).” PXY-1026, 5:55-60.</p> <p>The aqueous solution of Rice forms the colloidal suspension with iron and includes sodium sulfite.</p> <p>“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</p>

‘709 Patent Claims	Disclosure in Rice in view of Cantrell
	<p>approximately 8 to 12 mg/L per mg/L dissolved oxygen is suitable.” PXY-1006, 3:47-57. <i>See also</i> Claims 1, 3, and 4.</p>
<p>[12.0] 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.</p>	<p>Rice in view of Cantrell discloses this claim element.</p> <p>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.</p> <p>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.</p>
<p>[14.0] the method of claim 1 wherein said metal is iron.</p>	<p>Rice in view of Cantrell discloses this claim element.</p> <p>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.</p> <p>Specifically Cantrell describes “[w]hen zero-valent iron metal particles are used as the solid particles, suspensions of Fe<sup>0</sup> colloids (2±1 μm) are injected at a colloid injection rate, P≤0.01, and concentration, P~0.05, to uniformly distribute colloids throughout soil columns at concentrations &gt;0.3% (w/w).” PXY-1026, 5:55-60.</p>
<p>[15.0] The method of</p>	<p>Rice in view of Cantrell discloses this claim element.</p>

‘709 Patent Claims	Disclosure in Rice in view of Cantrell
claim 1 wherein said metal is in a colloidal suspension.	<p>Cantrell discloses a method to dechlorinate dissolved chlorinated solvents in ground water which includes the introduction of zero valent metal in a colloidal suspension into the soil pathways. PXY-1026, 2:11-19; 5:17-35, Claims 1, 13.</p> <p>Specifically Cantrell describes “[w]hen zero-valent iron metal particles are used as the solid particles, suspensions of Fe<sup>0</sup> colloids (2±1 μm) are injected at a colloid injection rate, P≤0.01, and concentration, P~0.05, to uniformly distribute colloids throughout soil columns at concentrations &gt;0.3% (w/w).” PXY-1026, 5:55-60.</p>
[16.0] The method of claim 15 wherein the colloidal suspension includes a reducing agent.	<p>Rice in view of Cantrell discloses this claim element.</p> <p>The aqueous solution of Rice which forms the fluid of the colloidal suspension contains sodium sulfite.</p> <p>“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, 3:47-57. <i>See also</i> Claims 1, 3, and 4.</p> <p>Sodium sulfite is a reducing agent. PXY-1006, 3:47-57.</p>

**XII. [Intentionally Left Blank]**

**A. [Intentionally Left Blank]**

115. [Intentionally Left Blank]

116. [Intentionally Left Blank]

117. [Intentionally Left Blank]

118. [Intentionally Left Blank]

**B. [Intentionally Left Blank]**

119. [Intentionally Left Blank]

120. [Intentionally Left Blank]

121. [Intentionally Left Blank]

**C. [Intentionally Left Blank]**

122. [Intentionally Left Blank]

**XIII. Challenge #5: Claims 4, 10, and 13 are obvious over Vance in view of Schuring**

**A. Vance in View of Schuring Teaches Every Element of Claims 4, 10, and 13**

123. As described in Section IX.A above, 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.

Specifically, Vance describes injecting under pressure a colloid suspension made up of elemental iron and a carbohydrate into the soil. PXY-1010, Claims 11-20.

124. Schuring discloses an apparatus and methods of pneumatic fracturing to increase soil permeability. Schuring discusses widespread contamination of soil and groundwater with “volatile organic compounds (VOC)” and notes that “technology is being developed for the in situ removal of VOC from the vadose zone. Such treatment technologies include vapor extraction, biodegradation, soil washing and thermal treatment.” PXY-1024, 1:42-48.

125. Schuring acknowledges the role of indigenous microorganisms in biodegrading VOCs and efforts to enhance favorable microorganism behavior:

As discussed above, biodegradation is another process which has effectively been used in the treatment of soils contaminated with hazardous organic compounds. Specifically, with biodegradation or bioremediation, the environmental conditions in the soil are altered to enhance microbial catabolism or to cometabolise the organic contaminant, thus transforming it into a simpler, non-toxic product. In most applications, indigenous microorganisms are utilized, although seeding of the soil with indigenous microorganisms has also been used where natural occurring organisms are unable to degrade the contaminants. PXY-1024, 2:57-68.

wells are drilled into the soil and nutrients for feeding the microbes are dropped down into the well, or microbes are seeded in the well. PXY-1024, 3:11-13.

126. The increased soil permeability created by the invention disclosed in Schuring improves the effectiveness of decontamination efforts. Schuring

recognizes “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.

127. Schuring continues:

In accordance with the present invention, by creating fissures and cracks in contaminated soil 16, a fractured soil formation 26 is formed so as to enhance the aforementioned remedial methods.

The principal effect that pneumatic fracturing has on the structure of soil 16 is that it establishes conductive channels 26, as shown in FIG. 4, which permit more rapid movement of liquid and vapor through the soil matrix. This is beneficial since essentially any treatment technology is limited by the permeability of the soil formation being treated. Thus, pneumatic fracturing according to the present invention reduces the treatment time and makes it feasible to treat tightly packed or extremely fine grained soils for which existing technologies are presently ineffective.

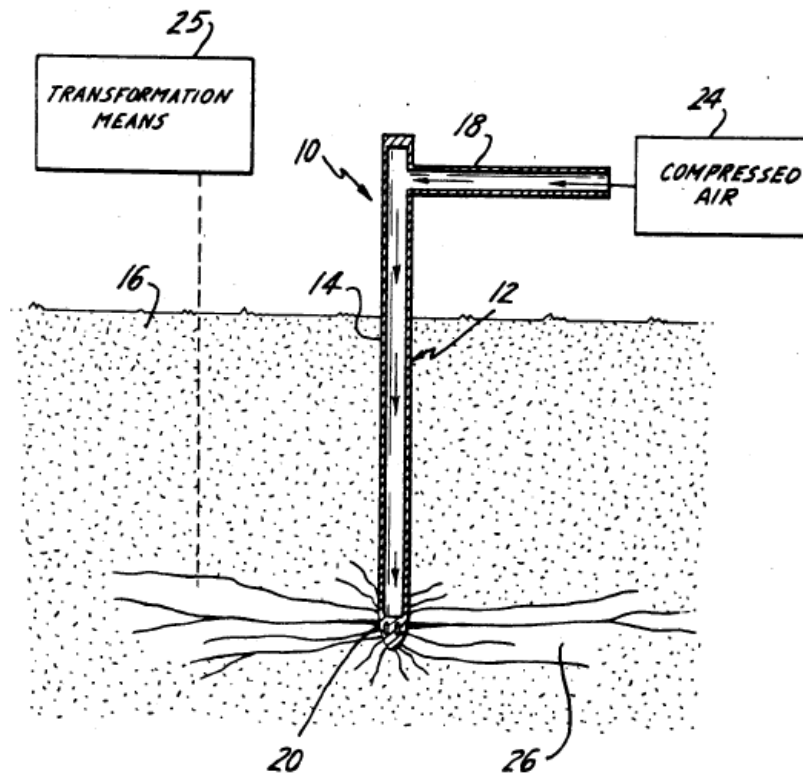
The primary transport mechanism for contaminant removal in pneumatically fractured soils will be vapor diffusion and flux out of the soil pores and into the secondary fractures and channels 28. The sudden air surge during the pneumatic fracturing procedure will also impart energy to the soil to break the surface tension of the contaminants adhering to the soil particles.

Pneumatic fracturing will also enhance biodegradation since it will modify the three important factors which affect the growth of subsurface microbial populations, namely oxygen concentration, permeability and water content. An additional benefit of the process for biodegradation is that it will increase the effectiveness of air spargers commonly used in such systems. PXY-1024, 9:12-38



128. The injection apparatus of Schuring is shown in Figure 1:

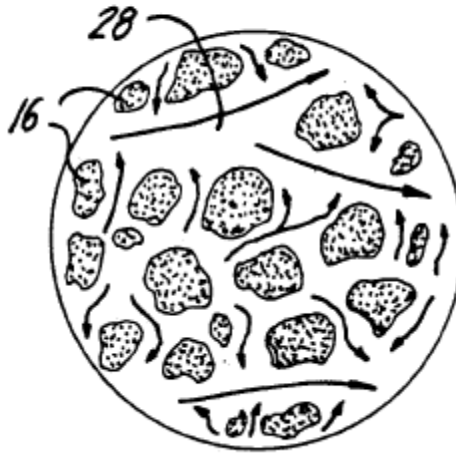
**FIG. 1**



PXY-1024, Fig. 1.

129. Figure 4 of Schuring illustrates “an enlarged elevational view of pneumatically fractured soil according to the present invention, showing vapor movement in the soil microstructure”, PXY-1024, 8:21-23:

**FIG. 4**



PXY-1024, Fig. 4.

**B. Motivation to Combine the References**

130. 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

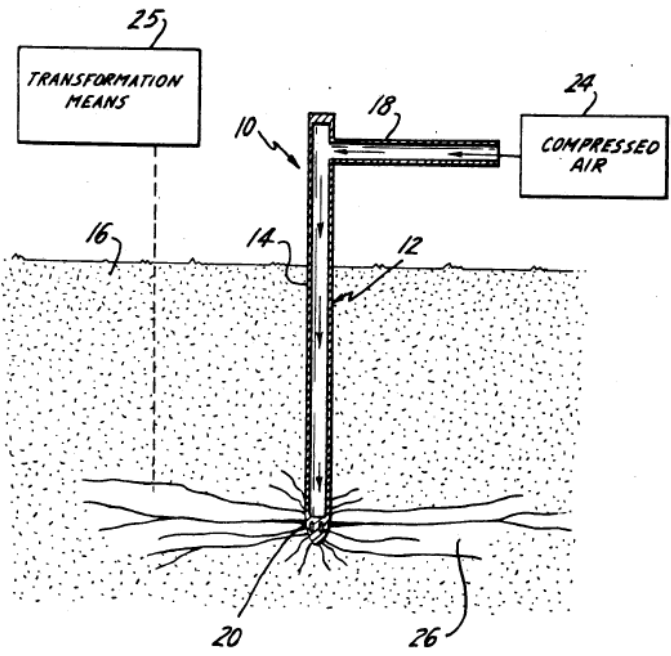
slurry within the subsurface over significant horizontal distances and 6) the modification would reduce the number of injection wells required.

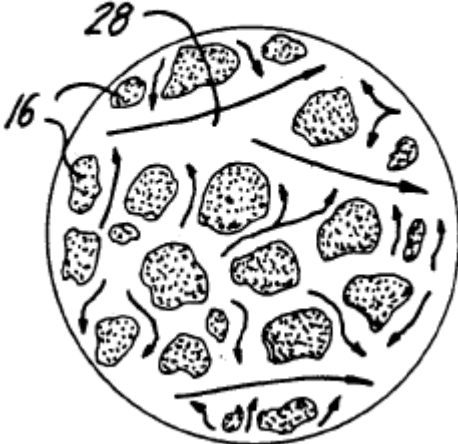
131. The proposed combination of Vance and Schuring would result in the claimed invention of Claims 4, 10, and 13.

### C. Detailed Invalidity Analysis

132. The following table explains how Vance in view of Schuring teaches Claims 4, 10, and 13 of the '709 Patent, alone or in combination with the knowledge, skill, and creativity of a POSA.

'709 Patent Claims	Disclosure in Vance in view of Schuring
[4.0] 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.	<p>Vance in view of Schuring discloses this claim element.</p> <p>Vance discloses the method of Claim 3. <i>See</i> Section IX.B above.</p> <p>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 that pneumatic fracturing “introduces flow channels into the soil” which enables distribution of the remediation treatments. PXY-1024, 5:32</p> <p>Schuring further explains:</p> <p>“The principal effect that pneumatic fracturing has on the structure of soil 16 is that it establishes conductive channels 26, as shown in FIG. 4, which permit more rapid movement of liquid and vapor through the soil</p>

'709 Patent Claims	Disclosure in Vance in view of Schuring
	<p>matrix. This is beneficial since essentially any treatment technology is limited by the permeability of the soil formation being treated. Thus, pneumatic fracturing according to the present invention reduces the treatment time and makes it feasible to treat tightly packed or extremely fine grained soils for which existing technologies are presently ineffective.” PXY-1024, 9:16-26.</p> <p style="text-align: center;"><b>FIG. 1</b></p>  <p>PXY-1024, Figure 1.</p>

'709 Patent Claims	Disclosure in Vance in view of Schuring
	<p style="text-align: center;"><b>FIG. 4</b></p>  <p>PXY-1024, Figure 4.</p>
<p>[10.0] The method of claim 4 wherein said gas is from the group of nitrogen and carbon dioxide.</p>	<p>Vance in view of Schuring discloses this claim element.</p> <p>The pneumatic fracturing taught by Schuring utilizes air as the gas:</p> <p>“Accordingly, high pressure air is injected into soil 16 to fracture the contaminated soil formation in order to accelerate the removal or treatment of hazardous liquids and vapors.” PXY-1024, 8:66-9:1. Air comprises nitrogen and carbon dioxide.</p> <p>Moreover, given that 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</p>

‘709 Patent Claims	Disclosure in Vance in view of Schuring
	separate pneumatic system would not be required.
[13.0] The method of claim 4 further including a final step of gas injection to clear said injection rod and fluid conduit lines connected thereto.	<p>Vance in view of Schuring discloses this claim element.</p> <p>Neither Schuring nor Vance disclose purging the injection rod or connected lines as a final step. However, cleaning equipment after use is well known in the art and is considered good practice. Further, ensuring all the remediation treatment is supplied into the soil is economic common sense. It is further understood by a POSA that purging the injection rod and conduit lines with the injection of gas a well-known and successful cleaning method.</p> <p>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. A POSA would be motivated to add the step to clean the equipment and supply all the slurry into the soil requiring remediation.</p>

#### **XIV. Challenge #6: Claim 8 is obvious over Hitchens in view of Grasso**

##### **A. Hitchens in View of Grasso Teaches Every Element of Claim 8**

133. Hitchens in view of Grasso discloses the claimed subject matter of Claim 8.

134. Hitchens is described above in Section VIII.A. As described above, Hitchens discloses “a method for enhancing the biodegradation of chlorinated

organic compounds in the ground by naturally occurring bacteria.” PXY-1003, 1:10-15. Hitchens discloses providing nutrients in the mixture as described in claim 7 (*See* VI.B.3.a.). Hitchens does not describe the use of ammonia and orthophosphate.

135. Grasso describes introducing nutrients such as ammonia and orthophosphate can “enhance the rate and extent of biodegradation”. PXY-1014 5.1.4.1.

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. PXY-1014 5.1.4.1

#### **B. Motivation to Combine Hitchens and Grasso**

136. 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 often the limiting factor in the subsurface environment and must be supplied to ensure biodegradation.

137. The proposed combination of Hitchens in view of Grasso would result in the claimed invention of Claim 8.

### C. Detailed Invalidity Analysis

138. The following table explains how Hitchens in view of Grasso teaches every element of Claim 8 of the ‘709 Patent, alone or in combination with the knowledge, skill, and creativity of a POSA.

‘709 Patent Claims	Disclosure in Hitchens in view of Grasso
<p>[8.0] The method of claim 7 wherein said nutrients are organic ammonia and ortho-phosphate.</p>	<p>Hitchens in view of Grasso discloses this claim element.</p> <p>Hitchens discloses the method of claim 7. <i>See</i> Section VIII, above. Hitchens does not expressly describe the nutrients are ammonia and orthophosphate.</p> <p>Grasso describes that introducing nutrients such as ammonia and orthophosphate can “enhance the rate and extent of biodegradation”. PXY-1014 5.1.4.1.</p> <p>“Phosphorus is added in the form of inorganic orthophosphate” PXY-1014 5.1.4.1</p> <p>“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.” PXY-1014 5.1.4.1</p> <p>“When nitrate is supplied as the only nitrogen source, it must be first reduced to ammonia before the bacterium can use it.” PXY-1014 5.1.4.1</p>



**XV. Challenge #7: Claim 6 is obvious over Hitchens in view of Odom**

**A. Hitchens in View of Odom Teaches Every Element of Claim 6**

139. Hitchens in view of Odom discloses the claimed subject matter of Claim 6.

140. Hitchens is described above in Section VIII.A and discloses the method of Claim 1 as shown above in Section VIII.B. Hitchens recognizes the dechlorination properties of B12:

vitamin B<sub>12</sub> (Co-containing porphyrin) which is often found in bacteria, and coenzyme F<sub>430</sub> (Ni-containing porphyrin), found only in methanogenic bacteria have the capacity to mediate the eight electron sequential reduction of PCE to ETH. PXY-1003, 2:63-67.

141. However, Hitchens does not explicitly disclose supplying B2 and B12 into the soil:

142. Odom is directed to a dehalogenation method:

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.

143. Odom discloses that the introduction of both vitamin B2 and vitamin B12 into soil and groundwater greatly accelerates dechlorination. PXY-1025, 15:65-67 (“dechlorination of CCl<sub>4</sub> to CHCl<sub>3</sub> was greatly accelerated when both riboflavin and a tetrapyrrole were present in the reaction mixture.”); 15:65-67

(“Dechlorination at levels 30-100 fold higher than hydrogenase-C3 were observed when both riboflavin and the tetrapyrrole were present.”).

144. Odom refers to vitamin B2 as “riboflavin” and vitamin B12 as “a tetrapyrrole” such as “cyanocobalamin”. *See* PXY-1025,15:30-16:15.

145. Odom discloses that the reagents will be introduced at the injection well in the same manner as the nutrients:

The present invention additionally provides a method of in situ remediation of organohalogen contaminated soil and groundwater. Conventional technology for managing groundwater which has become contaminated with pollutants such as organohalogens may be categorized into four categories: (1) containment of the contaminants using either physical barriers (i.e., slurry cutoff walls or sheet pilings) or hydraulic barriers created by a system of injection and pumping wells ... PXY-1025, 12:27-35.

Introduction of the nutritional amendment into the aquifer may be performed by pumping the nutrients into the aquifer via a series of wells placed upstream of the contamination plume and natural aquifer flow. PXY-1025, 22:50-53. *See also* PXY-1025, 22:13-23:27.

#### **B. Motivation to Combine Hitchens and Vance**

146. 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. 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.

147. The proposed combination of Hitchens in view of Odom would result in the claimed invention of Claim 6.

### C. Detailed Invalidity Analysis

148. The following table explains how Hitchens in view of Odom teaches every element of Claim 6 of the '709 Patent, alone or in combination with the knowledge, skill, and creativity of a POSA.

'709 Patent Claims	Disclosure in Hitchens in view of Odom
[6.0] The method of claim 1 wherein said organic hydrogen donor further includes vitamins B2 and B12.	<p>Hitchens in view of Odom discloses this claim element.</p> <p>Hitchens discloses the method of claim 1. <i>See</i> Section VIII, above.</p> <p>Odom discloses that the introduction of both vitamin B2 (riboflavin) and vitamin B12 (a tetrapyrrole) into the soil greatly accelerated dechlorination:</p> <p>“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.</p> <p>Odom discloses that the reagents will be introduced at the injection well in the same manner as the nutrients:</p> <p>“Dechlorination at levels 30-100 fold higher than hydrogenase-C3 were observed when both riboflavin and the tetrapyrrole were present.” PXY-1025, 16:7-</p>

‘709 Patent Claims	Disclosure in Hitchens in view of Odom
	<p>10.</p> <p>Riboflavin is vitamin B2 and the tetrapyrrole in Odom is described as cyanocobalamin or vitamin B12. <i>See</i> PXY-1025,15:30-16:15.</p>

## **XVI. Conclusion and Declaration**

**A.** The findings and opinions set forth in this declaration are based on my work and examinations to date. I may continue my examinations. I may also receive additional documentation and other factual evidence over the course of this litigation that will allow me to supplement and/or refine my opinions. I reserve the right to add to, alter, or delete my opinions and my declaration upon discovery of any additional information. I reserve the right to make such changes as may be deemed necessary.

**B.** In signing this declaration, I recognize that the declaration will be filed as evidence in a contested case before the Patent Trial and Appeal Board of the United States Patent and Trademark Office. I also recognize that I may be subject to cross-examination in the case and that cross-examination will take place within the United States. If cross-examination is required of me, I will appear for cross-examination within the United States during the time allotted for cross-examination.

**C.** I declare that all statements made herein on my own knowledge are true and that all statements made on information and belief are believed to be true, and further, that these statements were made with the knowledge that willful false

statements and the like so made are punishable by fine or imprisonment, or both,  
under Section 1001 of Title 18 of the United States Code.

Executed on: 11/24/2015

By: John Bergendahl  
Dr. John A. Bergendahl