UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

PHARAOH ENERGY SERVICES, LLC
Petitioner

v.

FLEX-CHEM HOLDING COMPANY, LLC
Patent Owner

Case No. IPR2024-00815
Patent No. 9,944,843
Issued: April 17, 2018
Application No.: 14/686,237
Filed: April 14, 2015

Title: STIMULATION OF WELLS IN NANO-DARCY SHALE FORMATIONS

PETITION FOR *INTER PARTES* REVIEW OF U.S. PATENT NO. 9,944,843 (CLAIMS 1–13)

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EXHIBIT LIST

Exhibit No.	Description
1001	U.S. Patent No. 9,944,843
1002	Prosecution History of U.S. Patent No. 9,944,843
1003	Expert Declaration of Dr. Jeffrey H. Harwell
1004	United States Patent No. 6,436,880 B1 to Frenier
1005	United States Patent No. 10,072,205 B2 to Reyes et al.
1006	Proof of Service of Summons and Complaint, *Flex-Chem Holding Company LLC v. Pharaoh Energy Services, LLC*, U.S. District Court for the Western District of Oklahoma, Case No. 5:23-cv-00316-JD
1007	U.S. Provisional Application No. 61/979,210 filed April 14, 2014
1008	U.S. Provisional Application No. 62/087,899 filed December 5, 2014
1009	Flex-Chem Claim Construction Brief, *Flex-Chem Holding Company LLC v. Pharaoh Energy Services, LLC*, U.S. District Court for the Western District of Oklahoma, Case No. 5:23-cv-00316-JD

1010	U.S. Patent. No. 1,877,504A to Grebe, entitled "Treatment of Deep Wells," issued September 13, 1932
1011	U.S. Patent. No. 2,094,479 to Vandergrift, entitled "Treatment of Wells," issued September 28, 1937
1012	U.S. Patent. No. 4,695,389 to Kubala entitled "AQUEOUS GELLING AND/OR FOAMING AGENTS FOR AQUEOUS ACIDS AND METHODS OF USING THE SAME," issued September 22, 1987
1013	U.S. Patent No. 4,574,050 to Crowe et al., entitled "METHOD FOR PREVENTING THE PRECIPITATION OF FERRIC COMPOUNDS DURING THE ACID TREATMENT OF WELLS," issued March 4, 1986
1014	U.S. Pat. No. 5,203,413 to Zerhboub, entitled "Product and Process for Acid Diversion in the Treatment of Subterranean Formations," issued April 20, 1993
1015	Borchardt, *Chemicals Used in Oil Field Operations*, 1989 (clean copy obtained April 16, 2024)
1016	Schlumberger Online Glossary, (screenshots compiled April 16, 2014)

I. INTRODUCTION

Pharaoh Energy Services, LLC ("Petitioner") petitions for *inter partes* review under 35 U.S.C. §§ 311–319 and 37 C.F.R. § 42.100 *et seq.* of all claims (1–13) of U.S. Patent No. 9,944,843 ("the '843 Patent") (Ex. 1001) (the "Challenged Claims"). This Petition sets forth two grounds for invalidity, which are likely to prevail based on the accompanying evidence, and as such, this Petition should be granted and the Challenged Claims canceled.

II. MANDATORY NOTICES UNDER 37 C.F.R. § 42.8

A. Real Parties-in-Interest Under 37 C.F.R. § 42.8(b)(1)

Other than Petitioner Pharaoh Energy Services, LLC, there are no other real parties-in-interest.

B. Related Matters Under 37 C.F.R. § 42.8(b)(2)

The '843 Patent is the subject of a patent infringement lawsuit brought by the Patent Owner Flex-Chem Holding Company, LLC ("Patent Owner") and its purported exclusive licensee Flex-Chem Services Corp. against Petitioner in the U.S. District Court for the Western District of Oklahoma, Case No. 5:23-cv-00316-JD ("Related Litigation"). Petitioner was served a copy of the Summons and Complaint on April 18, 2023. (A copy of the Proof of Service of the Summons and Complaint filed by Patent Owner in the Related Litigation is attached as Exhibit 1006).

C. Lead and Back-Up Counsel Under 37 C.F.R. § 42.8(b)(3)

Petitioner provides the following designation of counsel:

Lead Counsel	**Back-Up Counsel**
Jim V. John (Reg. No. 81,378) jjohn@ryanwhaley.com	Evan W. Talley, *Pro Hac Vice* anticipated etalley@ryanwhaley.com Elizabeth L. Isaac (Reg. No. 74,561) eisaac@ryanwhaley.com Jacob J. Oliphant (Reg. No. 81,369) joliphant@ryanwhaley.com
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D. Service Information Under 37 C.F.R. § 42.8(b)(4)

Petitioner may be served at the addresses shown above. Petitioner consents to electronic service: iprfilings@ryanwhaley.com. Pursuant to 37 C.F.R. § 42.10(b), filed with this Petition is a Power of Attorney.

III. PAYMENT OF FEES PURSUANT TO 37 C.F.R. §§ 42.15 AND 42.103

Per 37 C.F.R. §42.103, the Office is authorized to charge $41,500 and any additional fees to Deposit Account No. 606468.

IV. GROUNDS FOR STANDING PURSUANT TO 37 C.F.R. § 42.104(A)

Petitioner certifies that the '843 Patent is available for *inter partes* review and that Petitioner is not barred or estopped from requesting *inter partes* review challenging the claims of the '843 Patent on the grounds identified herein. Neither Petitioner, nor any party in privity with Petitioner: (1) has filed a civil action challenging the validity of the Challenged Claims of the '843 Patent; (ii) has been served a complaint alleging infringement of the '843 Patent more than one year prior to the present date; or (iii) is estopped from challenging the claims on the grounds identified in the Petition. The Challenged Claims also have not been the subject of a prior *inter partes* review or a finally concluded district court litigation.

V. IDENTIFICATION OF CHALLENGE PURSUANT TO 37 C.F.R. § 42.104(B) AND STATEMENT OF RELIEF REQUESTED

A. Claims for Which Review is Requested

Petitioner respectfully requests review and cancellation of claims 1–13 of the '843 Patent as unpatentable.

B. Citation of Prior Art

United States Patent No. 6,436,880 B1 to Frenier **("Frenier")** (Ex. 1004).

Frenier was filed on May 3, 2000 as U.S. Patent Application No. 09/563,557 and issued as U.S. Patent No. 6,436,880 B1 on August 20, 2002. Frenier qualifies as prior art under 35 U.S.C. § 102(a)(1).

United States Patent No. 10,072,205 B2 to Reyes et al. **("Reyes")** (Ex. 1005). Reyes was filed as U.S. Patent Application No. 13/925,888 on June 25, 2013. Reyes qualifies as prior art to the '843 Patent under 35 U.S.C. § 102(a)(2).

C. Statutory Grounds of Challenge

Petitioner asks the Board to institute an *inter partes* review under 35 U.S.C. § 314(a) as there is a reasonable likelihood that at least one or more of the Challenged Claims should be canceled as unpatentable on the following evidence and grounds:

Ground	Basis	Claims	References
1.	§ 102	1, 2, 4, 5, 7, and 13	Frenier
2.	§ 103	1–13	Frenier alone and/or in view of Reyes

For each of these grounds, in Section VII, the Petition demonstrates at least a reasonable likelihood, based on a preponderance of the evidence (35 U.S.C. § 316(e)), that Petitioner will prevail in establishing that at least one or more of the Challenged Claims are unpatentable. The Petitioner's arguments are supported by a Declaration from Jeffrey H. Harwell (Ex. 1003), Professor Emeritus at the University of Oklahoma, where he taught chemical engineering for nearly forty (40) years.

D. This Petition Presents Art and Arguments Not Previously Considered by the Office

Following the Board's precedential decision in *Advanced Bionics, LLC v. Med-El Elektromedizinische Gerate GmbH*, IPR2019-01469, Paper 6 (PTAB Feb. 13, 2020), the Board uses a two-part framework to analyze whether a Petition presents "the same or substantially the same prior art or arguments previously were presented to the Office:"

> (1) [determining] whether the same or substantially the same art previously was presented to the Office or whether the same or substantially the same arguments previously were presented to the Office; and (2) if either condition of [the] first part of the framework is satisfied, [determining] whether the petitioner has demonstrated that the Office erred in a manner material to the patentability of challenged claims.

Advanced Bionics, Paper 6 at 8. In analyzing whether the same or substantially the same art or arguments were previously presented to the Office, the Board considers various factors, including: (i) the similarities and material differences between the asserted art and the prior art previously presented to the Office; (ii) the cumulative nature of the asserted art and the prior art previously evaluated by the Office; and (iii) the extent of the overlap between the arguments made before the Office and the manner in which Petitioner relies on the prior art or the patent owner distinguishes the prior art. *Id.* at 8–10; *see also Becton, Dickinson & Co. v. Braun Melsungen AG*,

IPR2017-01586, Paper 8, 17–18 (Dec. 15, 2015).

In determining whether Petitioner has demonstrated that the Office erred in a manner material to patentability of the challenged claims, the Board considers various factors, including: (iv) the extent to which the asserted art was evaluated by the Office, including whether the prior art was the basis for rejection during examination; (v) whether Petitioner has pointed out sufficiently how the Office erred in its evaluation of the asserted prior art; and (vi) the extent to which additional evidence and facts presented in the petition warrant reconsideration of prior art or documents. *Advanced Bionics*, 8–10; *Becton-Dickinson*, 17–18.

Of the two prior art references upon which Petitioner bases its grounds of challenge, only Frenier was "considered" during prosecution of the '843 Patent. Even then, it was only included on an information disclosure statement ("IDS") submitted by Patent Owner (Ex. 1002, at p. 328), which included 21 other issued U.S. Patents, 15 U.S. Patent Publications, two foreign patent documents, and 13 non-patent literature documents. *Id.* at 328–333.[1] While the Examiner noted she

[1] The IDSs submitted by Patent Owner include three other Frenier prior art references, a non-patent literature document entitled, "Use of Highly Acid-Soluble Chelating Agents in Well Stimulation Services" (Ex. 1002, at p. 315) and two U.S. Patents, Nos. 7,192,908 and 8,312,929 (*id.* at p. 329). While the examiner indicated she "considered" the three additional Frenier references (*id.* at pp. 266, 270), none were the basis for a rejection or otherwise cited during prosecution of the '843 Patent. Most notably, none of these other three Frenier references are included in the grounds presented in this Petition.

"considered" Frenier by not lining through it on the Examiner-signed copy of the IDS (*id.* at 270), Frenier did not serve as the basis for any rejection, nor was it otherwise cited to or addressed during prosecution of the '843 Patent.

While several other "Reyes" references were included in IDSs during prosecution of the '843 Patent (*see, e.g. id.* at pp. 330–31), as well as an International Search Report (*id.* at 320) and Written Opinion of the International Search Authority (*id.* at 338–341), the Reyes prior art reference relied on this Petition was not cited in any IDS, considered, addressed, or the basis for any rejection during prosecution of the '843 Patent.

Even if the inclusion of Frenier in an IDS and the Examiner's not lining through it means that Frenier was "considered" during prosecution of the '843 Patent, the Examiner erred in a manner material to the patentability of the Challenged Claims by not ascertaining that Frenier included the very limitations the Examiner believed put the Challenged Claims in shape for allowance. It appears the Examiner felt inclusion in Claim 1 of the terms "existing" (before "well in a nano-darcy shale formation" in the preamble) and "exits the well and" (between "treatment mixture" and "contacts the nano-darcy shale formation" in the "injecting" step) were enough to differentiate the Challenged Claims from the prior art relied on by the Examiner. *Id.* at pp. 87, 92–94. Demonstrated below, Frenier includes both of these limitations, and if properly "considered" by the Examiner, would have

precluded the Challenged Claims from issuance.

With regard to inclusion of the "treatment mixture ***exits the well and*** contacts the nano-darcy shale formation" limitation, Frenier discloses that "matrix acidizing methods generally involve pumping the acid-containing well treatment composition down the wellbore and out through perforations into the target formation." Ex. 1004, at 7:24–27. Based on this disclosure in Frenier, Dr. Harwell opines a person of ordinary skill in the art would have understood to "inject[] the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture exits the well and contacts the nano-darcy shale formation." Ex. 1003, at ¶¶ 106–111.

With regard to inclusion of "existing" before "well in a nano-darcy shale formation" in the preamble of Claim 1, Frenier describes that "the present invention relates to the use of acidic fluids in stimulation and workover operation." Ex. 1004, at 4:9–10. As Dr. Harwell opines, a person of ordinary skill in the art would have understood the inclusion of "workover operations" to mean that Frenier applies "to stimulating or otherwise treating pre-existing wells, in addition to new wells." Ex. 1003, at ¶ 78–79. Dr. Harwell further cites to industry literature to support that "a 'workover operation' has generally meant '[t]he repair or **stimulation of an existing production well** for the purpose of restoring, prolonging or enhancing the production of hydrocarbons.'" *Id.* at ¶ 80.

Applying factors (iv)–(vi) of the *Advanced Bionics* and *Becton-Dickinson* framework leads only to the conclusion that the Examiner erred in its treatment of Frenier. Although Frenier was "considered," it did not serve as the basis for any rejection, despite its explicit disclosure of the two limitations that the Examiner felt put the '843 Patent in position for allowance. Petitioner has also presented the declaration and opinion of Dr. Harwell—which was not before the Examiner during prosecution of the '843—to provide additional support and context for what a person of ordinary skill in the art would have understood those disclosures to mean. The Examiner's overlooking of Frenier's explicit disclosures and additional evidence presented through the Declaration of Dr. Harwell. *See, e.g., DraftKings Inc. v. Interactive Games*, IPR2020-01107, Paper 10 (PTAB Jan. 6, 2021) (finding that Examiner erred in a manner material to the patentability of the challenged claims where the primary reference for each presented ground was included in an IDS and acknowledged by the Examiner during prosecution but did not serve as the basis for a rejection and where the Petitioner presented additional evidence in the form of an expert declaration and testimony that was not before the Examiner during prosecution).

E. There Have Been No Other Petitions Filed

None of the Challenged Claims have been the subject of any other Petition for *inter partes* review.

F. Person of Ordinary Skill in the Art

Patent Owner has described a person of ordinary skill in the art ("POSITA") at the time of the alleged invention of the '843 Patent had at least a bachelor's degree in chemistry, chemical engineering, petroleum engineering, geology, or the equivalent, and at least two years of experience in oil and natural gas well treatments, or an equivalent amount of relevant work and/or educational experience. Although Petitioner does not fully concur, for the purposes of this Petition, Petitioner is prepared to adopt the level of ordinary skill in the art set forth by Patent Owner.

Dr. Jeffrey Harwell, PH.D., meets these criteria and concurrently submits a Declaration (Ex. 1003) under 37 C.F.R. § 1.68 in support of this petition.

G. Supporting Evidence Under 37 C.F.R. § 42.104(b)(5)

An Exhibit List identifying the exhibits relied upon is included, at p. iv, *supra*.

VI. SUMMARY OF THE '843 PATENT

A. Effective Filing Date of the '843 Patent

The '843 Patent issued on April 17, 2018, from U.S Patent Application No. 14/686,237, filed April 14, 2015 ("the '237 Application"). The '843 Patent claims priority to U.S. Provisional Application No. 61/979,210 filed April 14, 2014 ("the '210 Provisional") (Ex. 1007) and to U.S. Provisional Application No. 62/087,899 filed December 5, 2014 ("the '899 Provisional") (Ex. 1008). Therefore, solely for the purposes of this filing, the earliest effective filing date of the '843 Patent is April

14, 2014.

B. The Specification

The specification of the ’843 Patent describes “formulations and methods for stimulating the production from wells in nano-darcy shale formations” generally including the following steps:

> (i) providing a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10;
>
> (ii) injecting the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture contacts the nano-darcy shale formation;
>
> (iii) maintaining the treatment mixture in contact with the nano-darcy shale formation for a contact time of between about 1 minute to 100 days, thereby allowing the metal complexing agent to bind with at least some naturally-occurring metals contained within the nano-darcy shale formation; and
>
> (iv) removing the treatment mixture from the well after the contact time, thereby removing the bound naturally-occurring metals from the non-darcy shale formation and thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method.

Ex. 1001, 2:35–53.[2] The specification of the ’843 Patent further defines what chemicals may be a “metal complexing agent”:

> For the purposes of this disclosure, a metal-complexing agent may be any chemical that can bind with a metal regardless of the binding mechanism and includes **sequestration agent**s, reducing agents,

[2] The notation N:XX–YY refers to column (or page) number N, and lines XX to YY of a reference.

> **chelating agents**, ligands porphyrins, pigments, peptides, saccharides and/or nucleic acids. In some embodiments, the metal-complexing agent is a **chelating agent**, an alkali metal salt thereof, a non-alkali metal salt thereof, or any combination thereof may be included in the treatment fluids described herein.

Id. at 4:54–63 (emphasis added). The most notable of these are chelating and sequestering agents because, as shown below, they have been used (and described for use) for the methods claimed in the '843 Patent prior to its earliest effective filing date. The specification of the '843 Patent further states that "the well stimulation mixture can contain the metal complexing agent as well as multiple chemical additives as desired." *Id*. at 6:26–30; 6:42; 7:3, 7:21; 7:36; and 7:49–50. These additives too were used and described for use for the claimed methods of the '843 Patent prior to April 14, 2014.

While the '843 Patent does not explicitly refer to it as such, the specification of the '843 Patent describes what has been known in the industry for more than seventy-five years as "matrix acidizing." Methods of matrix acidizing generally involve "pumping the acid-containing well treatment composition down the wellbore and out through perforations into the target formation… After the composition has been injected into the formation, optionally the well can be shut in for a period of time to allow more complete reaction between the acid and the formation material." Ex. 1004 at 7:24–34. "The desired result of the treatment is an increase in the permeability of the formation, for example by the creation or

enlargement of passageways through the formation, and therefore an increase in the rate of production of formation fluids such as oil and gas." *Id.* at 7:34–38.

C. The '843 Patent's Prosecution History

The '237 Application that became the '843 Patent was filed on April 14, 2015 with two independent claims and seventeen total claims (Ex. 1002 at pp. 385–88). The '237 Application claimed priority to the '210 Provisional and the '899 Provisional. *Id.* at 396.

On July 28, 2017, the Office issued a Non-Final Rejection rejecting all of the pending claims: Claims 1–7 and 10–13 were rejected as under 35 U.S.C. § 103 as being unpatentable over Horton et al. (US 2011/0053811 A1) ("Horton"). *Id.* at 244–50. Claims 8–9 were rejected as under 35 U.S.C. § 103 as being unpatentable over Horton in view of Conway et al. (US 2015/0114653 A1) ("Conway"). *Id.* at 250–52.

Patent Owner initiated an interview with the Examiner, which took place on September 16, 2017. *Id.* at 191. The Applicant-Initiated Interview Summary identifies Horton as the "prior art discussed" and states, "[t]he attorney pointed out that in the present invention, it is important for the treatment to penetrate the shale formation to change the characteristics of the formation, whereas Horton teaches an additive that prevents the fluid from entering the formation." *Id.*. However, "[t]he Examiner noted that it is important that such a limitation of the fluid leaving the wellbore and entering the formation would only be persuasive if it were recited in

the claim language." *Id.*

On October 24, 2017, Patent Owner responded to the rejections over Horton by amending claim 1 as follows: "injecting the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture exits the well and contacts the nano-darcy shale formation…." *Id.* at 87. Patent Owner further argued that "Horton teaches exactly the opposite: Horton does not want any remediation mixture to leave the well and contact the formation." *Id.* at 93. With respect to claim 8–9, Patent Owner responded that Conway "does not qualify as prior art" because "the instant application has an effective filing date more than one year prior to the publication of Conway" and "Conway and the instant application have a common inventor, Bryce Conway, and a common assignee, Flex-Chem." *Id.* at 94.

The Examiner issued a Notice of Allowance on March 14, 2018. *Id.* at 7–8.

D. How the Challenged Claim(s) are To Be Construed under 37 C.F.R. § 42.104(b)(3)

In the Related Litigation, Patent Owner has proposed construing claim terms of the '843 Patent using their plain and ordinary meaning to a POSITA. *See*, Ex. 1009. Petitioner does not necessarily agree that a POSITA in April 2014 would have construed the claim terms as proposed by Patent Owner. However, for purposes of this Petition (and any subsequent PTAB proceedings), Petitioner adopts and applies

the plain and ordinary meaning of the claim terms.

VII. DETAILED EXPLANATION OF GROUNDS FOR CANCELLATION OF THE CHALLENGED CLAIMS

A. Ground I: Claims 1, 2, 4, 5, 7, and 13 are Anticipated under 35 U.S.C. § 102 by Frenier

i. Overview of Frenier

Frenier, entitled "Well Treatment Fluids Comprising Chelating Agents," is directed to "stimulation of hydrocarbon wells and in particular to acid fluids and methods of using such fluids in treating a subterranean formation having low permeability." Ex. 1004, at 1:6–9. Frenier discloses several methods of "acid-treating a subterranean formation." *Id*. at 4:45. Generally, the methods include "the step of injecting a well treatment fluid composition via a wellbore into a subterranean formation… at a fluid pressure that is less than the minimum in situ rock stress (i.e., a matrix acidizing method), [but] can also be performed at a higher pressure (i.e., an acid fracturing method)." *Id.* at 4:49–53. Frenier explains that the practice of using treatment fluids to improve the productivity of oil wells was disclosed over a century ago. *See id.* at 1:66–2:3.

Frenier identifies the need for "an acid well treatment fluid that is relatively inexpensive and can readily control precipitation of iron." *Id.* at 3:54–56. Moreover, Frenier states it would be "desirable" if an "acid well treatment fluid could lead to improved radial penetration than is commonly seen for acid well treatment fluids

known in the art," "that the acid well treatment fluid could be used in either matrix acidizing or fracture acidizing treatments," and "for the acid well treatment fluid to be useful in control of scale, such as alkaline earth carbonate scales in a wellbore in a carbonate formation." *Id.* at 3:57–65. Frenier addresses these needs by disclosing "the use of acidic fluids in stimulation and workover operations, and in particular, in matrix acidizing or fracture acidizing treatments." *Id.* at 4:8–12. Indeed, "[t]he desired result of the treatment is an increase in the permeability of the formation, for example by the creation or enlargement of passageways through the formation, and therefore an increase in the rate of production of formation fluids such as oil and gas." *Id.* at 7:34–38.

ii. Anticipation by Frenier

"When, as by a recitation of ranges or otherwise, a claim covers several compositions, the claim is 'anticipated' if one of them is in the prior art." *Titanium Metals Corp. v. Banner*, 778 F.2d 775, 227 USPQ 773 (Fed. Cir. 1985). When a patent claims a range, as in this case, that range is anticipated by a prior art reference if the reference discloses a point within the range. *Id*. If the prior art discloses its own range, rather than a specific point, then the prior art is only anticipatory if it describes the claimed range with sufficient specificity such that a reasonable fact finder could conclude that there is no reasonable difference in how the invention operates over the ranges. *Ineos USA LLC v. Berry Plastics Corp.*, 783 F.3d 865, 869

(Fed. Cir. 2015). When the prior art discloses a range which touches or overlaps the claimed range, but no specific examples falling within the claimed range are disclosed, the reference anticipates the claims if the claimed subject matter is disclosed in the reference with "sufficient specificity." *See* MPEP 2131.03.

For a disclosure to be implicit or inherent it must be a necessary feature or result of an embodiment sufficiently described and enabled in the prior art reference. However, it is not necessary that a POSITA would have recognized that implicit or inherent disclosure at the time of invention. *See*, *e.g.*, *MEHL/Biophile Int'l Corp. v. Milgraum*, 192 F.3d 1362, 1365 (Fed. Cir. 1999). If the claimed composition is identical to a composition disclosed by a prior art reference, it does not matter that the applicant recognized additional properties of that composition. *See Titanium Metals Corp. of America v. Banner*, 778 F.2d at 782 (finding that the prior art anticipated the claims at issue despite the alleged newly-discovered property of the claimed composition).

When a reference or material from various documents is explicitly incorporated by reference, it is "effectively part of the host document as if [it] were explicitly contained therein." *Arbutus Biopharma Corp. v. ModernaTX, Inc.*, 65 F.4th 656, 662–63 (Fed. Cir. 2023). While looking at the reference as a whole, the Board will "conclude whether or not that reference discloses all elements of the claimed invention arranged as in the claim." *Arbutus*, 65 F.4th at 662 (citing *Net*

MoneyIN, Inc. v. VeriSign, Inc., 545 F.3d 1359, 1369 n.5 (Fed. Cir. 2008)).

1. Independent Claim 1

a. Element 1[Preamble]: "A method for stimulating an existing well in a nano-darcy shale formation comprising:"

Frenier states "[t]his invention relates to the stimulation of hydrocarbon wells and in particular to acid fluids and methods of using such fluids in **treating a subterranean formation having low permeability**." Ex. 1004 at 1:6-9 (emphasis added). Frenier further explains that "[t]echniques performed by hydrocarbon producers to increase the net permeability of the reservoir are referred to as 'stimulation.' Essentially, one can perform a stimulation technique by… (2) injecting chemicals through the wellbore and into the formation to react with and dissolve small portions of the formation to create alternative flowpaths for the hydrocarbon…." *Id*. at 1:39–54. A POSITA would have known and understood Frenier to apply to "subterranean formation[s] having low permeability," including nano-darcy shale formations. Ex. 1003 at ¶¶ 167–69.

To the extent the claimed invention is limited to existing wells, Frenier discloses that its "invention relates to the use of acidic fluids in stimulation and **workover operations**, and in particular, in matrix acidizing or fracture acidizing treatments." Ex. 1004 at 4:8–12 (emphasis added). A POSITA would have understood Frenier's disclosure to apply to stimulating existing wells in addition to new wells. Ex. 1003 at ¶ 79.

Accordingly, to the extent the preamble of Claim 1 is limiting, Frenier discloses each limitation.

b. Element 1[a]: "providing a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10;"

Frenier discloses a "well treatment fluid," i.e., a treatment mixture, comprising: "a first acid; water; and a hydroxyethylaminocarboxylic acid." Ex. 1004 at 4:12–13. Frenier further explains that "[e]xamples of first acids include non-oxidizing mineral acids such as hydrochloric acid, sulfuric acid, phosphoric acid, hydrofluoric acid, and mixtures thereof. Other examples include non-oxidizing organic acids such as formic acid, acetic acid, and mixtures thereof." *Id*. at 5:27–31. However, "hydroxyethylaminocarboxylic acids have the advantage of being effective in acidizing treatment of a formation **without the use of a first acid.**" *Id*. at 6:31–33 (emphasis added). "Preferably, the hydroxyethylaminocarboxylic acid is selected from hydroxyethylethylenediaminetriacetic acid (HEDTA), hydroxyethyliminodiacetic acid (HEIDA), or a mixture thereof." *Id*. at 4:13–17. Thus, Frenier discloses various treatment mixtures wherein the metal complexing agent may be a traditional chelating or sequestering agent such as HEDTA or HEIDA, or may be a lower pH mineral or organic acid such as HCL, HF, or acetic acid. A POSITA would have known and understood the same. Ex. 1003 at ¶ 90.

Frenier provides examples of "typical concentration ranges" for its treatment

mixture with and without a first acid, including between 1% and 30% by weight metal complexing agent, which covers much of the claimed range set forth in Element1[a]. Ex. 1004 at 6:25–47. Frenier further discloses treatment mixtures having between 15% and 28% by weight metal complexing agent and between 10% and 30% by weight metal complexing agent. *See* claims 1, 9, and 28 at Ex. 1004, 11:27–34; 11:54–63; 13:3–20. However, Frenier includes that "[t]he proportion of the various components of a composition of the present invention will vary depending on the characteristics of the formation to be treated, the acid to be used, and other factors well known in the art." *Id*. at 6:16–19.

Frenier further discloses, with sufficient specificity, the treatment mixture having "a pH of between about 0 and 10" as required by Element 1[a]. Specifically, claim 28 of Frenier discloses the treatment mixture comprising "a pH controller agent to adjust the pH of the treatment fluid between to about 1 to about 12." *Id*. at 13:9–10. Frenier also discloses an embodiment containing water and a hydroxyethylaminocarboxylic acid, where "the pH of the well treatment fluid is less than about 12, more preferably from about 1 to about 4, and can be controlled by an organic acid, a mineral acid, or a base." *Id*. at 4:57–60. Additionally, Frenier cites Athey et al., U.S. Pat. No. 5,972,868, entitled "Method for Controlling Alkaline Earth and Transition Metal Scaling with 2-Hydroxyethyl Iminodiacetic Acid," which, according to Frenier, discloses "compositions comprising 2-

hydroxyethyliminodiacetic acid as a chelant for the removal of alkaline earth scale in downhole equipment. The compositions can be at any pH from about 2 to about 13." *Id*. at 3:66-67; 4:1-5.

Thus, Frenier discloses all of the limitations of this claim element. A POSITA would have understood to provide a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10 to stimulate a subterranean formation. Ex. 1003 at ¶ 102.

c. Element 1[b]: "injecting the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture exits the well and contacts the nano-darcy shale formation;"

Frenier discloses "a method of acid-treating a subterranean formation." Ex. 1004 at 4:44–45. "The method includes the step of injecting a well treatment fluid composition via a wellbore **into a subterranean formation**." *Id*. at 4:45–47 (emphasis added). Indeed, "the well treatment fluid composition comprises the components described above." *Id*. at 4:47–49. Moreover, "the injection step is **preferably performed at a fluid pressure that is less than the minimum in situ rock stress (i.e., a matrix acidizing method)**, [but] the method can also be performed at a higher pressure (i.e., an acid fracturing method)." *Id*. at 4:49–53 (emphasis added). Claims 9 and 28 of Frenier are also directed to these "acid-treatment" and "matrix acidizing" methods. *Id*. at 11:54–62. Frenier discloses that

"[s]uch matrix acidizing methods generally involve pumping the acid-containing well treatment composition down the wellbore and **out through perforations into the target formation**." *Id*. at 7:24–27 (emphasis added).

Thus, all of the limitations of claim Element 1[b] are taught by Frenier.

d. Element 1[c]: "maintaining the treatment mixture in contact with the nano-darcy shale formation for a contact time of between about 1 minute to 100 days, thereby allowing the metal complexing agent to bind with at least some naturally-occurring metals contained within the nano-darcy shale formation;"

Frenier discloses that "[a]fter the composition has been injected into the formation, optionally the well can be shut in for a period of time to allow more complete reaction between the acid and the formation material." *Id.* at 7:30–34. Frenier also explains that "[p]arameters such as pumping rate, pumping time, **shut-in time**, acid content, and additive package, must be determined for each particular treatment since each of these parameters depends upon the extent of the damage, formation geology (e.g., permeability), formation temperature, depth of the producing zone, etc. A well-treatment designer of ordinary skill is familiar with the essential features of matrix acidizing treatments." *Id*. at 7:39–46 (emphasis added). Notably, Frenier explicitly provides examples wherein the treatment solutions were maintained in contact with the formation samples for 24 and 72 hours. *Id.* at 9:27–66.

A contact time between about 1 minute to 100 days would have been inherent to a POSITA as a period to allow for a proper reaction between the metal complexing agent and the metals of the formation so as to avoid precipitation of the metals and congestion of the well. Ex. 1003 at ¶ 115.

Frenier also discloses the limitation that the metal complexing agent binds "with at least some naturally-occurring metals contained within the nano-darcy shale formation." Specifically, claim 28 of Frenier includes the element: "said matrix-acidizing resulting in the formation of wormholes due to the dissolution of minerals **naturally-present in the formation**." Ex. 1004 at 13:17–19 (emphasis added).

Frenier also provides that "[u]se of the well treatment compositions of the present invention in a matrix acidizing process provides for the ready control of iron liberated from the formation, thus helping to minimize the precipitation of iron in the formation or in the wellbore." *Id*. at 8:12–16. Additionally, Frenier states, "types of scales treatable by the fluids and techniques of the present invention include chloride scales (e.g., NaCl), iron scales (e.g., FeS, Fe2O3), silica scales (e.g., SiO2), sulfate scales (e.g. CaSO4), and hydroxide scales (e.g., Mg(OH)2)" in addition to carbonate scales such as CaCO3 and FeCO3. *Id*. at 7:1–11. Frenier also provides examples of its inventive concepts wherein "[i]t was observed that substantially all the Fe and Ca present in the sample comprising Na3HEDTA remained in solution (i.e. both metals were fully sequestered by Na3HEDTA) at both room temperature

and 150° F…" and the results "indicate[d] HEDTA and HEIDA were effective at sequestering iron and minimizing its precipitation." *Id*. at 8:62–65; 9:54–55.

Therefore, Frenier discloses "allowing the metal complexing agent to bind with at least some naturally-occurring metals contained within the nano-darcy shale formation." Ex. 1003 at ¶ 121.

Frenier discloses all of the limitations of Element 1[c]. Ex. 1003 at ¶ 123.

e. Element 1[d]: "removing the treatment mixture from the well after the contact time, thereby removing the bound naturally-occurring metals from the non-darcy shale formation and thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method."

Frenier discloses removal of the treatment mixture from the well, thereby removing the metals from the formation and improving the hydrocarbon production from the well in several different instances. First, Frenier plainly states, "the present invention relates to methods to **enhance the productivity of hydrocarbon wells** (e.g., oil wells) by creating alternate flowpaths by removing portions of a wellbore coating, dissolving small portions of the formation, **or removing (by dissolution) near-wellbore formation damage**." Ex. 1004 at 1:55–60. "The efficiency of a matrix treatment depends primarily upon **removing** or bypassing regions of low permeability that restrict productivity." *Id*. at 6:49–51 (emphasis added).

Second, Frenier states, "[a]nother aspect of the present invention is a method

of acid-treating a subterranean formation. The method includes the step of injecting a well treatment fluid composition via a wellbore into a subterranean formation… **The method can also be used to remove deposits from the wellbore**." *Id.* at 4:53–55.

Third, Frenier also teaches using the well treatment fluid to remove drilling mud that contains naturally-occurring metals:

> The well treatment fluid compositions of the present invention can be used to remove drilling mud from the wellbore. Removal of drilling mud occurs especially readily if the mud contains carbonates, especially calcium carbonate. Removal of drilling mud can be performed by any technique know [*sic*] in the art, and involves the steps of injecting a well treatment fluid composition of the present invention into the wellbore.

Id. at 7:13–20. Fourth, Frenier incorporates by reference, U.S. Patent. No. 1,877,504A to Grebe, entitled "Treatment of Deep Wells," issued September 13, 1932 ("Grebe") (Ex. 1010). Grebe is directed to "increasing or restoring" the flow of an oil well in a limestone formation "by treating with a quantity of an acid, such as hydrochloric acid. The acid has the effect of attacking and dissolving the rock, thereby enlarging the cavity at the bottom of the well, or the channels and pores in the rock through which oil flows to the well." Ex. 1010 at page 1, lines 45–52. Grebe further discloses, "[i]n carrying out our improved method we employ a mineral acid, preferably hydrochloric acid, inasmuch as the latter **upon reacting with the calcareous rock forms water soluble salts which remain in**

solution and are removed from the well with the spent acid." *Id*. at lines 59–74 (emphasis added).

Fifth, Frenier also incorporates by reference U.S. Patent. No. 2,094,479 to Vandergrift, entitled "Treatment of Wells," issued September 28, 1937 ("Vandergrift"). Vandergrift is directed to a "method of increasing the flow of fluid mineral products from a well which comprises introducing into the well a chemical reagent comprising hydrofluoric acid and a mineral acid and permitting it to act upon the formations adjacent to the well cavity." Ex. 1011 at 4:14–19. Vandergrift discloses, "[a]fter the mixed acid reagent has had sufficient time to act upon the formation, **the spent acid may be removed by pumping or bailing out**. It may sometimes be desirable to flush out the well with water or other neutral liquid after the spent acid has been removed and before the well is put back into production." *Id*. at 4:34–40 (emphasis added).

Sixth, Frenier incorporates by reference, U.S. Patent. No. 4,695,389 to Kubala entitled "Aqueous Gelling And/Or Foaming Agents For Aqueous Acids And Methods Of Using The Same," issued September 22, 1987 ("Kubala") (Ex. 1012). Kubala is directed to:

> [A] novel acid gelling agent that can be combined with aqueous acids at higher concentrations of the acids to produce highly retarded gelled acid systems that exhibit the unique characteristic of building viscosity during the carbonate rock dissolving process, reaching a peak viscosity when the acid is partially spent (e.g., approximately 12% HCl and in the presence of calcium ion) with virtually complete

breaking of the gel when the acid is totally spent.

Ex. 12 at 2:6–14. Kubala further discloses, "after the reaction is completed, the viscosity again drops to a very low value insuring **ease of removal and backflushing of the formation**." *Id*. at 10:43–46 (emphasis added).

Accordingly, all of the limitations of this claim element are disclosed by Frenier and its incorporated references. A POSITA would have known and understood, in light of Frenier and the references incorporated therein, to "remov[e] the treatment mixture from the well after the contact time**,** thereby removing the bound naturally-occurring metals from the non-darcy shale formation and thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method." Ex. 1003 at ¶ 134.

Frenier fully disclosed each and every element of Claims 1 of the '843 Patent to a POSITA prior to April 14, 2014. Ex. 1003 at ¶ 136.

2. Dependent Claim 2

a. "The method of claim 1 wherein the metal complexing agent is citric acid."

Frenier cites to U.S. Pat. No. 4,888,121 to Dill, entitled "Compositions and Method for Controlling Precipitation When Acidizing Sour Wells" ("Dill"). According to Frenier, Dill "discloses an acidizing composition that includes an acid such as CHI; **an iron sequestering agent such as citric acid**, EDTA, or nitrilotriacetic acid (NTA); and a sulfide modifier such as formaldehyde." *Id*. at

3:39–45 (emphasis added).

Thus, a POSITA would have known and understood, or at a minimum it would have been obvious, to use citric acid as the metal complexing/sequestering agent prior to April 2014. Ex. 1003 at ¶ 139.

Additionally, Frenier discloses, "[i]t has been discovered that hydroxyethylaminocarboxylic acids are very soluble in mineral acids or **organic acids**, and can be mixed with mineral acids or organic acids to provide a wide range of useful formulations." Ex. 1004 at 5:16–20 (emphasis added). A POSITA would have known and understood Frenier's disclosure of "organic acids" as metal complexing agents to include citric acid.

Frenier also incorporates by reference U.S. Patent No. 4,574,050 to Crowe et al., entitled "METHOD FOR PREVENTING THE PRECIPITATION OF FERRIC COMPOUNDS DURING THE ACID TREATMENT OF WELLS," issued March 4, 1986 ("Crowe") (Ex. 1013). Crowe is directed to "methods of substantially eliminating problems arising from the presence of ferric ions which occur during the acidization of wells." Ex. 1013 at 1:15–17. Crowe states, "[t]he most common iron sequestering agents in commercial practice are **citric acid**, ethylenediaminetetra acetic acid (EDTA), nitrilotriacetic acid (NTA), acetic acid, lactic acid, and **citric acid/acetic acid mixtures**." *Id*. at 1:58–64 (emphasis added).

A POSITA would have known and understood Frenier's disclosure of

"organic acids" as metal complexing agents to include citric acid. *Id*. It was also well-within the knowledge of a POSITA prior to April 14, 2014 to select citric acid as a chelating agent for well treatment operations and compositions. Ex. 1003 at ¶ 145. Thus, Frenier discloses every element of dependent claim 2.

3. Dependent Claim 4

a. "The method of claim 1 wherein the metal complexing agent is acetic acid."

As set forth above, Frenier discloses that "[e]xamples of first acids include non-oxidizing mineral acids such as hydrochloric acid, sulfuric acid, phosphoric acid, hydrofluoric acid, and mixtures thereof. Other examples include non-oxidizing organic acids such as formic acid, **acetic acid**, and mixtures thereof." *Id*. at 5:27–31 (emphasis added).

Thus, a POSITA would have known in light of Frenier and its incorporated references to use acetic acid as a metal complexing agent. Ex. 1003 at ¶ 148. It was also well-within the knowledge of a POSITA prior to April 14, 2014 to use acetic acid as a complexing agent. Ex. 1003 at ¶ 148.

4. Dependent Claim 5

a. "The method of claim 1 wherein the metal complexing agent includes at least one of ethylenediaminetetraacetic acid (EDTA), propylenediaminetetraacetic acid (PDTA), nitrilotriacetic acid (NTA), N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), diethylenetriaminepentaacetic acid (DTPA), hydroxyethyliminodiacetic acid (HEIDA),

> **cyclohexylenediaminetetraacetic acid (CDTA), diphenylaminesulfonic acid (DPAS), ethylenediaminedi(o-hydroxyphenylacetic) acid (EDDHA), glucoheptonic acid, gluconic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, phthalic acid, terephthalic acid, aconitic acid, carballylic acid, trimesic acid, isocitric acid, citric acid, or any salt or derivative of any of the previously listed compounds."**

As set forth above, Frenier discloses the metal complexing agent including several, if not all, of the acids listed in claim 5. Ex. 1003 at ¶ 151. For example, Frenier discloses selecting HEDTA or HEIDA as the metal complexing agent. Ex. 1004 at 4:13–17.

Additionally, Petitioner incorporates herein the statements and arguments above for Dependent Claims 2 with respect to the metal complexing agent including citric acid.

With respect to any remaining limitations, Petitioner notes they are part of a Markush element and, thus, anticipation only requires that any one of the listed acids is disclosed by the prior art. *See Fresenius USA*, 582 F.3d at 1298.

Accordingly, Frenier discloses every element of Dependent Claim 5.

5. Dependent Claim 7

a. "The method of claim 1 wherein the treatment mixture further comprises between 0.1 and 95% of acid(s) as pH modifier."

Frenier discloses a treatment mixture including the use of additives, explicitly including pH modifiers: "[t]he composition can also include one or more additives

that are compatible with the acid composition, such as… **pH control agents**. Many such additives are well known in the art." Ex. 1004 at 5:56–63 (emphasis added).

Indeed, the use of pH modifiers to control the pH of the treatment mixtures, and the performance of the treatment mixture, was well known and understood by POSITAs prior to April 14, 2014. Ex. 1003 at ¶ 157. Frenier provides examples of treatment mixtures comprising 0.1% to 15% by weight pH modifier (*id*. at 6:40–45), and claims 28, 31, 49 are directed to treatment mixtures that include pH modifiers. *Id*. at 13:3–19; 13:25–27; 14:44–45.

In light of Frenier, a POSITA would have known to include between .0 and 95% of acid(s) as pH modifier. Ex. 1003 at ¶ 160. Thus, Frenier discloses every limitation of dependent claim 7.

6. Dependent Claim 13

a. "The method of claim 1 further wherein the metal complexing agent is selected from acetic acid, acrylates, dihydroxymaleic acid, salts of dihydroxymaleic acid, EDTA (ethylenediamine tetraacetic acid), salts of EDTA, erythorbic acid, erythroboric acid, formic acid, gluconodeltalactone, GLDA (glutamic acid N,N-diacetic acid), salts of GLDA, HEDTA (hydroxyethylenediamine triacetic acid), salts of HEDTA, HEIDA (disodium ethanoldiglycine), salts of HEIDA, MGDA (methylglycine N,N-diacetic acid), salts of MGDA, NTA (nitriolotriacetic acid), organic metal complexers, ligands, porphyrins, pigments, peptides, saccharides, nucleic acids, phosphonic acid, polyacrylic acid and citric acid in an amount sufficient to sequester at least a portion of a metal compound."

As set forth above, Frenier discloses the metal complexing agent selected from several, if not all, of the acids listed in claim 13. For example, Frenier discloses selecting HEDTA or HEIDA as the metal complexing agent. Ex. 1004 at 4:13–17.

Additionally, Petitioner incorporates herein its statements and arguments from Dependent Claims 2 and 5.

With respect to any remaining limitations, Petitioner notes they are part of a Markush element and, thus, anticipation only requires that any one of the listed acids is disclosed by the prior art. *See Fresenius USA*, 582 F.3d at 1298.

Accordingly, Frenier discloses every element of Dependent Claim 13. Ex. 1003 at ¶ 165.

B. Ground II: Claims 1–13 are Obvious under 35 U.S.C. § 103(a) over Frenier alone and/or in combination with Reyes

i. Overview of Reyes

Reyes is directed to "acidizing subterranean formations, and, more specifically, to methods for acidizing subterranean formations in the presence of a chelating agent that is initially unable to complex a metal ion." Ex. 1005 at 1:8–12. When able, "chelating agents may be utilized in various acidizing operations or in other subterranean treatment operations to complex a metal ion and sequester it in the subterranean environment." *Id*. at 3:49–52. Reyes further discloses methods and systems for acidizing subterranean formations using treatment fluids containing "dual-functioning" chelating agents. That is, the "chelating agents may play a dual

role of slowing dissolution rates and performing a delayed metal complexation reaction." *Id*. at 5:2–4.

Reyes discloses that "[a]cidizing operations may be used to stimulate a subterranean formation to increase production of a hydrocarbon resource therefrom." *Id*. at 1:29–31. "Introduction of the acidizing fluid to the subterranean formation may take place at matrix flow rates without fracturing of the formation matrix, or at higher injection rates and pressures to fracture the formation (i.e., an acid-fracturing operation)." *Id*. at 1:31–35. "During an acidizing operation, an acid-soluble material in the subterranean formation can be dissolved by one or more acids to expand existing flow pathways in the subterranean formation, to create new flow pathways in the subterranean formation, and/or to remove acid-soluble precipitation damage in the subterranean formation." *Id*. at 1:35–41. "The acid-soluble material being dissolved by the acid(s) can be part of or formed from the native formation matrix or can have been deliberately introduced into the subterranean formation in conjunction with a stimulation or like treatment operation (e.g., proppant or gravel particulates)." *Id*. at 1:41–46.

Similarly to Frenier, Reyes also covers the formulation of treatment fluids with various additives commonly used in downhole operations to optimize the acidizing process. *Id*. at 11:34–54.

ii. Obviousness

A party that petitions the Board for a determination of obviousness must show that 'a skilled artisan would have been motivated to combine the teachings of the prior art references to achieve the claimed invention, and that the skilled artisan would have had a reasonable expectation of success in doing so.'" *Apotex Inc. v. Wyeth LLC*, IPR2014-00115, Paper 94, slip op. at 11 (Apr. 20, 2015) (quoting *Procter & Gamble Co. v. Teva Pharms. USA, Inc.*, 566 F.3d 989, 994 (Fed. Cir. 2009)). The obviousness determination turns on underlying factual inquiries involving: (1) the scope and content of prior art, (2) differences between claims and prior art, (3) the level of ordinary skill in pertinent art, and (4) secondary considerations such as commercial success and satisfaction of a long-felt need. *Procter & Gamble Co. v. Teva Pharm. USA, Inc.*, 566 F.3d 989, 994 (Fed. Cir. 2009) (quoting *Graham v. John Deere Co.,* 383 U.S. 1, 17, 86 S.Ct. 684, 15 L.Ed.2d 545 (1966)).

iii. Motivation to Combine Frenier and Reyes

To the extent needed, a POSITA would have been motivated to combine the teachings of Frenier and Reyes to produce obvious, beneficial, and predictable results. Frenier and Reyes are analogous prior art and both pertain to the same field of endeavor. Specifically, both seek to improve generally known stimulation, matrix acidizing, or acid-treating operations on subterranean formations using treatment mixtures that include chelating and/or sequestering agents (e.g., EDTA or HEDTA).

As discussed in both Frenier and Reyes, the beneficial results of the disclosed operations include increasing the permeability of subterranean formations of presently low permeability. Increasing the permeability of the formations provides the benefit of allowing more hydrocarbons to be recovered from the well. The optimized performance of hydrocarbon-bearing wells would have been desirable to a POSITA.

Both Frenier and Reyes identify significant limitations of traditional acid stimulation treatments. Both patents acknowledge the risk of precipitation damage resulting from interactions with the treatment fluid and metal ions within the formation. They both recognize concerns regarding the rapid dissolution of injected acids and their rapid reaction with formation materials. While both patents express concerns about the rapid reactions between acids and formation materials, Frenier emphasizes the limited radial penetration depth of acid treatments resulting from these reactions, whereas Reyes focuses on the broader issue of formation matrix erosion due to the same rapid reactions.

A POSITA, aware of these challenges, would recognize the potential benefit of combining these disclosures to address them simultaneously. Here, both patents propose using acids and chelating agents, albeit with potentially different formulations or specific functionalities. A POSITA would recognize that combining these approaches offers enhanced effectiveness, particularly with regards to

mitigating corrosion and precipitation damage using chelating agents. A POSITA would recognize that combining these acid formulations and chelating agents might lead to a synergistic effect. The particular acid formulations could potentially enhance the overall effectiveness of the chelating agents and minimize unwanted reactions or interactions within the formation.

Additionally, both inventions involve wellbore stimulation using a combination of acids and chelating agents. This shared technological base would make the combination readily apparent to a POSITA. A POSITA would identify no technical obstacles likely to impede this combination, as both approaches involve readily available wellbore treatment components. Moreover, the oil and gas industry has been constantly seeking to improve stimulation treatment efficiency while minimizing formation damage and equipment corrosion. A POSITA would recognize that combining these disclosures aligns with this trend toward more targeted and effective wellbore treatments.

Overall, the shared problem recognition, complementary solutions, potential for synergistic effects, and common technological foundation strongly suggest that a POSITA would have been motivated to combine the teachings of Frenier and Reyes. This combination offers a solution that addresses limitations identified in both disclosures, leading to potentially more effective and comprehensive wellbore stimulation treatments.

Further, given the similarity of the disclosures, a POSITA would have a reasonable expectation of success in combining the teachings of Frenier and Reyes. Specifically, for any treatment mixture compositions or methods of using those compositions not disclosed or rendered obvious by Frenier alone, a POSITA would have wanted to include the more-explicit teachings of Reyes (e.g., biodegradable chelating agents or adding biocides) to improve the treatment operation, thus improving hydrocarbon production, and would have had a reasonable expectation of doing so.

1. Independent Claim 1

a. Element 1[Preamble]: "A method for stimulating an existing well in a nano-darcy shale formation comprising:"

Petitioner incorporates herein its statements and arguments above with respect to Frenier's disclosure of Element 1[Preamble]. It would have been obvious to a POSITA to apply the disclosures of Frenier to stimulating existing wells in addition to new wells. Ex. 1003 at ¶ 171.

Moreover, Reyes states, "[t]he present disclosure generally relates to acidizing subterranean formations." Ex. 1005 at 3:44–45. "Acidizing operations may be used to stimulate a subterranean formation to increase production of a hydrocarbon resource therefrom." *Id.* at 1:29–31. Reyes further explains that "[i]n some embodiments, the treatment fluids may be used to treat an **existing fracture** in a

subterranean formation in order to enhance a flow pathway therein." *Id.* at 13:40–42 (emphasis added). *Id.* at 1:39–54.

Thus, to the extent the preamble of Claim 1 is limiting, it would have been obvious to a POSITA prior to the effective filing date of the '843 Patent to apply the stimulation methods of Frenier and Reyes to an existing well in a nano-darcy shale formation. Ex. 1003 at ¶ 175.

b. Element 1[a]: "providing a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10;"

Petitioner incorporates herein its statements and arguments above with respect to Frenier's anticipation of Element 1[a]. *See* Ex. 1004 at 4:12–17; 5:27–31; Ex. 1003 at ¶ 183. Frenier discloses treatment mixtures having properties within the ranges required by claim 1. Ex. 1004 at 6:25–47; 11:27–34; 11:54–63; 13:3–20; 4:57–60. Specific examples include treatment mixtures having 20% by weight metal complexing agent at pH values of 4, 2.5, and 3.5. *Id*. at 10:61–64.

Indeed, to the extent the ranges of the '843 Patent are not anticipated by Frenier, it would have been obvious to a POSITA prior to the effective filing date of the '843 Patent to provide a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10. Ex. 1003 at ¶ 191. As stated by the Examiner, "since it has been held that where the general conditions of a claim are disclosed in the prior art, discovering the optimum or

workable ranges involves only routine skill in the art." *In re Aller*, 105 USPQ 233." Ex. 1002 at 246.

Reyes discloses a "treatment fluid," i.e., a treatment mixture, comprising: "an aqueous carrier fluid, an acid, and a chelating agent…." Ex. 1005 at 6:6–8. Similarly to Frenier, either the chelating agent or the acid disclosed by Reyes teaches the metal complexing agent of the '843 patent. Reyes states, "the concentration of the chelating agent in the treatment fluid may range between about 0.1% to about 40% of the treatment fluid by weight." *Id.* at 11:21–23. Reyes also predominantly provides the example of using aminopolycarboxylic acid chelating agents but also states that, alternatively, "traditional chelating agents such as" EDTA, HEDTA, HEIDA, and citric acid may be used. *Id*. at 10:59–67; 11:1–4.

Moreover, Reyes states that "[o]ne of ordinary skill in the art will recognize that there are fairly well established pH ranges over which a chelating agent can be expected to effectively complex a metal ion." *Id*. at 4:5–16. Thus, to the extent the limitation "providing a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10" is not anticipated or rendered obvious by Frenier, such a limitation would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier and Reyes. Ex. 1003 at ¶¶ 196–97.

c. Element 1[b]: "injecting the treatment mixture into the well at a pressure less than a fracture pressure of

the nano-darcy shale formation until at least some of the treatment mixture exits the well and contacts the nano-darcy shale formation;"

Petitioner incorporates herein its statements and arguments above with respect to Frenier's disclosure of Element 1[b]. However, it at least would have been obvious to a POSITA prior to the effective filing date of the '843 Patent to "inject[] the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture exits the well and contacts the nano-darcy shale formation." Ex. 1003 at ¶ 201.

Additionally, Reyes discloses, "in some embodiments, the treatment fluids described herein can be introduced to a subterranean formation **below a fracture gradient pressure of the subterranean formation**." Ex. 1005 at 4:44–45 (emphasis added). Reyes additionally states, "[i]ntroduction of the acidizing fluid to the subterranean formation may take place at matrix flow rates without fracturing of the formation matrix…." *Id*. at 1:31–33.

Reyes also discloses that "[t]he treatment fluid may be conveyed via line 12 to wellhead 14, where the treatment fluid enters tubular 16, tubular 16 extending from wellhead 14 into subterranean formation 18. **Upon being ejected from tubular 16**, **the treatment fluid may subsequently penetrate into subterranean formation 18**." *Id*. at 12:46–50 (emphasis added). Thus, Reyes explicitly discloses all of the limitations of this claim element.

At a minimum, Element 1[b] would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier and Reyes. Ex. 1003 at ¶¶ 204–05.

d. Element 1[c]: "maintaining the treatment mixture in contact with the nano-darcy shale formation for a contact time of between about 1 minute to 100 days, thereby allowing the metal complexing agent to bind with at least some naturally-occurring metals contained within the nano-darcy shale formation;"

Petitioner incorporates herein its statements and arguments above with respect to Frenier's disclosure of Element 1[c]. Frenier discloses that "[a]fter the composition has been injected into the formation, optionally the well can be shut in for a period of time to allow more complete reaction between the acid and the formation material." Ex. 1004 at 7:30–34. Frenier also explains that "[p]arameters such as … **shut-in time** … must be determined for each particular treatment." *Id*. at 7:39–46 (emphasis added). Notably, in the examples provided in Frenier, the treatment solutions were maintained in contact with the formation samples for 24 and 72 hours. Ex. 1004 at 9:27–66.

Thus, a contact time of 1 minute to 100 days would have been obvious to a POSITA as a period to allow for a proper reaction between the metal complexing agent and the metals of the formation. Ex. 1003 at ¶ 208; *see also* Ex. 1002 at 246 (Examiner stating, "where the general conditions of a claim are disclosed in the prior art, discovering the optimum or workable ranges involves only routine skill in the

art. *In re Aller*, 105 USPQ 233.”).

Frenier discloses the limitation that the metal complexing agent binds “with at least some naturally-occurring metals contained within the nano-darcy shale formation.” Specifically, claim 28 of Frenier includes the element: “said matrix-acidizing resulting in the formation of wormholes due to the dissolution of minerals **naturally-present in the formation**.” *Id*. at 13:17–19 (emphasis added).

As shown above, Frenier provides several metals naturally present in formation for which its methods and compositions can be used to treat. *Id*. at 8:12–16; 7:1–11; 8:62–65; 9:54–55.

It would have been obvious to a POSITA in light of Frenier to maintain contact between the treatment mixture for some time between 1 minute and 100 days so that that metal complexing agent can react with at least some naturally occurring metals in the nano-darcy shale formation. Ex. 1003 at ¶ 216.

Additionally, Reyes states as follows:

> In various embodiments, the methods described herein may comprise complexing a metal ion with the chelating agent … The metal ion being complexed by the chelating agent is not believed to be particularly limited. **Illustrative sources of the metal ion may include, for example, a native carbonate mineral present in the subterranean formation** … **In various embodiments, the metal ion being complexed by the chelating agent may include, for example, a calcium ion, a magnesium ion, an iron ion, or any combination thereof.**

Ex. 1005 at 9:54-67; 10:1-13 (emphasis added). Thus, Reyes also discloses Element

1[c]. Therefore, this claim element would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier and Reyes. Ex. 1003 at ¶¶ 217–18.

e. Element 1[d]: "removing the treatment mixture from the well after the contact time, thereby removing the bound naturally-occurring metals from the non-darcy shale formation and thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method."

Petitioner incorporates herein its statements and arguments above with respect to Frenier's disclosure Element 1[d], and specifically the at least six instances in which Frenier teaches Element 1[d].

Additionally, it would have been obvious to a POSITA prior to the effective filing date of the '843 Patent to "remov[e] the treatment mixture from the well after the contact time, thereby removing the bound naturally-occurring metals from the non-darcy shale formation and thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method." Ex. 1003 at ¶ 227. A POSITA would have been motivated to remove the bound metals from the formation during this period to avoid precipitation of the metals, thereby congesting the well. *Id.* at ¶ 228.

Thus, each and every element of Claims 1 of the '843 Patent would have been obvious to a POSITA prior to its effective filing date. *Id.* at ¶ 229.

Additionally, Reyes states, "[a]cidizing operations may be used to **stimulate a subterranean formation to increase production of a hydrocarbon resource therefrom**." Ex. 1005 at 1:29–31 (emphasis added). Reyes further discloses that "the treatment fluid may, in some embodiments, flow back to wellhead 14 and exit subterranean formation 18. In some embodiments, **the treatment fluid that has flowed back to wellhead 14 may subsequently be recovered** and recirculated to subterranean formation 18." *Id*. at 12:62-67 (emphasis added). Therefore, this claim element would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier and Reyes. Ex. 1003 at ¶ 232.

2. Dependent Claim 2

a. "The method of claim 1 wherein the metal complexing agent is citric acid."

Petitioner incorporates herein its statements and arguments above with respect to Frenier's disclosure of dependent claim 2. Frenier cites to Dill, which discloses a composition including "**an iron sequestering agent such as citric acid.**" Ex. 1004 at 3:39–45 (emphasis added). at 1:15–17. Crowe similarly states, "[t]he most common iron sequestering agents in commercial practice are citric acid, ethylenediaminetetra acetic acid (EDTA), nitrilotriacetic acid (NTA), acetic acid, lactic acid, and citric acid/acetic acid mixtures." Ex. 1013 at 1:58–64 (emphasis added). Additionally, Frenier discloses the use of "organic acids" as metal complexing agents, which a POSITA would have understood to include citric acid.

Ex. 1004 st 5:16–20; Ex. 1003 at ¶¶ 235–36.

Thus, selecting citric acid to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier and its incorporated references.

Reyes explicitly discloses the use of "traditional chelating agents such as … **citric acid**." Ex. 1005 at 10:59–67; 11:1–4 (emphasis added).

Thus, selecting citric acid to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier and Reyes.

3. Dependent Claim 3

a. "The method of claim 1 wherein the metal complexing agent is ethylenediaminetetraacetic acid (EDTA)."

Petitioner incorporates herein its statements and arguments above with respect to Frenier's disclosure of dependent claim 2. *See* Ex. 1004 at 1:58–64 (Crowe disclosing EDTA as one of the most common iron sequestering agents). Notably, although it found HEDTA to perform better, Frenier also provides examples of using EDTA to sequester iron and minimize precipitation. *See id*. at 8:40–11:25.

Thus, selecting EDTA to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier. Ex. 1003 at ¶ 249.

Additionally, Reyes explicitly discloses, "traditional chelating agents such as

… **ethylenediaminetetraacetic acid (EDTA)** … may be included in the treatment fluids described herein." Ex. 1005 at 10:59–67; 11:1–4 (emphasis added).

Selecting EDTA to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier and Reyes. Ex. 1003 at ¶ 251.

4. Dependent Claim 4

a. "The method of claim 1 wherein the metal complexing agent is acetic acid."

Petitioner incorporates herein its statements and arguments above with respect to Frenier's disclosure of dependent claim 4. Frenier discloses the use of **acetic acid** as a metal complexing agent. Ex. 1004 at 5:27–31.

Additionally, Reyes explicitly discloses that "the treatment fluids described herein may include an acid…. Suitable organic acids may include, for example, formic acid, **acetic acid**, chloroacetic acid, dichloroacetic acid, trichloroacetic acid, methanesulfonic acid and the like." Ex. 1005 at 7:34–46 (emphasis added). Reyes further states, "[b]oth mineral acids (e.g., hydrochloric acid) and organic acids (e.g., **acetic** and formic acids) can be used to treat a carbonate formation, often with similar degrees of success." *Id.* at 1:59–62 (emphasis added).

Thus, selecting acetic acid to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier alone or with Reyes. Ex. 1003 at ¶ 253.

5. Dependent Claim 5

a. "The method of claim 1 wherein the metal complexing agent includes at least one of ethylenediaminetetraacetic acid (EDTA), propylenediaminetetraacetic acid (PDTA), nitrilotriacetic acid (NTA), N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), diethylenetriaminepentaacetic acid (DTPA), hydroxyethyliminodiacetic acid (HEIDA), cyclohexylenediaminetetraacetic acid (CDTA), diphenylaminesulfonic acid (DPAS), ethylenediaminedi(o-hydroxyphenylacetic) acid (EDDHA), glucoheptonic acid, gluconic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, phthalic acid, terephthalic acid, aconitic acid, carballylic acid, trimesic acid, isocitric acid, citric acid, or any salt or derivative of any of the previously listed compounds."

Petitioner incorporates herein its statements and arguments above with respect to Frenier's disclosure of dependent claim 5. Frenier discloses the metal complexing agent including several, if not all, of the acids listed in claim 5. For example, Frenier discloses selecting HEDTA or HEIDA as the metal complexing agent. Ex. 1004 at 4:13–17.

Reyes also discloses, notably in the same order as the '843 Patent, the metal complexing agent including several, if not all, of the acids listed in claim 5. Reyes states, "as an alternative to aminopolycarboxylic acid chelating agents, traditional chelating agents such as, for example, [**EDTA, PDTA, NTA, HEDTA, DTPA, HEIDA, CDTA, DPAS, EDDHA**], **glucoheptonic acid, gluconic acid, citric acid**,

any salt thereof, any derivative thereof, or the like may be included in the treatment fluids described herein." Ex. 1005 at 10:59–67; 11:1–4 (emphasis added).

Thus, selecting one of the acids listed in claim 5 to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier alone or in combination with Reyes. Ex. 1003 at ¶ 260.

6. Dependent Claim 6

a. "The method of claim 1 wherein the treatment mixture comprises: between about 0.1% and 95% by weight metal complexing agent;"

As set forth above in Petitioner's arguments addressing independent claim 1, Frenier provides examples of "typical concentration ranges" for its treatment mixture with and without a first acid, including between 1% and 30%, between 15% and 28%, and between 10% and 30% by weight metal complexing agent. *See* claims 1, 9, and 28 at Ex. 1004 at 6:25–47; 11:27–34; 11:54–63; 13:3–20.

Thus, the limitations of dependent claim 6 would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier. Ex. 1003 at ¶ 263.

Additionally, Reyes states, "the concentration of the chelating agent in the treatment fluid may range between about 0.1% to about 40% of the treatment fluid by weight." Ex. 1005 at 11:21–23. Reyes similarly discloses the ranges between

about 0.1% to about 5%, between about 1% to about 10%, between about 5% to about 15%, between about 0.5% to about 5%, between about 10% to about 25%, between about 10% to about 40%, and between about 30% to about 40% of the treatment fluid by weight. *Id*. at 11:23–33.

Indeed, to the extent the ranges of the '843 Patent are not anticipated by Frenier, it would have been obvious to a POSITA prior to the effective filing date of the '843 Patent, given Frenier alone or in combination with Reyes, to provide a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent. Ex. 1003 at ¶ 266. As stated by the Examiner (and is applicable to all of the forthcoming elements of claim 6), "since it has been held that where the general conditions of a claim are disclosed in the prior art, discovering the optimum or workable ranges involves only routine skill in the art. *In re Aller*, 105 USPQ 233." Ex. 1002 at 246.

b. "between 1 and 10,000 parts per million (ppm) of a corrosion inhibitor;"

Frenier discloses a treatment mixture including the use of additives, explicitly including corrosion inhibitors and examples thereof. Ex. 1004 at 5:56–65 (Frenier listing well known additives including corrosion inhibitors). Reyes also provides a non-exclusive list of additives that are beneficial and well known in the art, explicitly including corrosion inhibitors. Ex. 1005 at 11:34-54 (emphasis added).

Thus, it would have been obvious to a POSITA prior to the effective filing

date of the '843 Patent to provide a treatment mixture including between 1 and 10,000 parts per million (ppm) of a corrosion inhibitor. Ex. 1003 at ¶ 271.

c. "between 1 and 10,000 ppm of a biocide;"

Frenier discloses a treatment mixture including the use of additives, such as a biocide. Specifically, Frenier states, "[t]he composition can also include one or more additives… [and many] such additives are well known in the art." Ex. 1004 at 5:56–63. Indeed, the use of biocides in matrix acidizing treatment solutions was well known in the art prior to April 14, 2014, and a POSITA would have known, or at least it would have been obvious, to include such additives into the treatment mixture to control microbial growth. Ex. 1003 at ¶ 274.

Additionally, Reyes discloses a treatment mixture including the use of additives, such as a bactericide, i.e., a "biocide." Ex. 1005 at 11:34-54 (Reyes listing additives, including "bactericides")

It would have been known and understood, or at least obvious, to a POSITA prior to the effective filing date of the '843 Patent to provide a treatment mixture including between 1 and 10,000 parts per million (ppm) of a biocide. Ex. 1003 at ¶ 276.

d. "between 1 and 10,000 ppm of a colloidal silica deposition inhibitor;"

Frenier discloses a treatment mixture including the use of additives, such as a colloidal silica deposition inhibitor. Ex. 1004 at 5:56–63. Indeed, the use of colloidal

silica deposition inhibitors in matrix acidizing treatment solutions was well known in the art prior to April 14, 2014, and a POSITA would have known, or at least it would have been obvious, to include such additives into the treatment mixture to inhibit colloidal silica deposits. Ex. 1003 at ¶ 278.

Reyes discloses a treatment mixture including the use of additives, such as silica scale control additives, i.e., "a colloidal silica deposition inhibitor." Ex. 1005 at 11:34-54 (Reyes listing additives, including "silica scale control additives").

Thus, it would have been obvious to a POSITA prior to the effective filing date of the '843 Patent to provide a treatment mixture including between 1 and 10,000 parts per million (ppm) of a colloidal silica deposition inhibitor. Ex. 1003 at ¶ 280.

e. "between 1 and 500 gallons per thousand gallons (gpt) of a mutual solvent; "

Frenier discloses a treatment mixture including the use of additives, explicitly including solvents: Ex. 1004 at 5:56–63 (Frenier listing additives, including "solvents").

Reyes discloses a treatment mixture including the use of additives, explicitly including various types of mutual solvents, such as "hydrate inhibitors." Reyes states, "the treatment fluids described herein may further comprise any number of additives that are commonly used in downhole operations including, for example, … **hydrate inhibitors** … [O]ne of ordinary skill in the art will be able to formulate

a treatment fluid having properties suitable for a given application." Ex. 1005 at 11:34-54 (emphasis added).

It would have been obvious to a POSITA prior to the effective filing date of the '843 Patent to provide a treatment mixture including between 1 and 500 gallons per thousand gallons (gpt) of a mutual solvent in light of Frenier alone or in combination with Reyes. Ex. 1003 at ¶¶ 282–84.

f. "and between 1 and 2000 parts per million of a surfactant."

Frenier discloses a treatment mixture including the use of additives, explicitly including surfactants. Ex. 1004 at 5:56–63 (Frenier listing additives, including "surfactants").

Reyes also discloses a treatment mixture including the use of additives, explicitly including surfactants. Ex. 1005 at 11:34-54 (Reyes listing additives, including surfactants).

It would have been obvious to a POSITA prior to the effective filing date of the '843 Patent to provide a treatment mixture including between 1 and 2000 parts per million of a surfactant. Ex. 1003 at ¶ 287.

7. Dependent Claim 7

a. "The method of claim 1 wherein the treatment mixture further comprises between 0.1 and 95% of acid(s) as pH modifier."

Petitioner incorporates herein its statements and arguments above with respect

to Frenier's disclosure of dependent claim 7. Frenier discloses the use of "pH control agents." Ex. 1004 at 5:56–63 (emphasis added).

Additionally, Frenier provides examples of treatment mixtures comprising 0.1% to 15% by weight pH modifier (*id*. at 6:40–45), and claims 28, 31, 49 are directed to treatment mixtures that include pH modifiers. *Id*. at 13:3–19; 13:25–27; 14:44–45.

Reyes also discloses a treatment mixture including the use of additives, explicitly including "**pH control agents (e.g., buffers).**" Ex. 1005 at 11:34-54 (emphasis added).

It would have been obvious to a POSITA prior to the effective filing date of the '843 Patent to provide a treatment mixture further comprising between 0.1 and 95% of acid(s) as pH modifier. Ex. 1003 at ¶¶ 290–91. As stated by the Examiner, "since it has been held that where the general conditions of a claim are disclosed in the prior art, discovering the optimum or workable ranges involves only routine skill in the art. *In re Aller*, 105 USPQ 233." Ex. 1002 at 246.

8. Dependent Claim 8

a. "The method of claim 1 wherein the injecting operation further comprises: alternately injecting a first amount of treatment mixture and a second amount of a diverting mixture into the well."

Frenier discloses the use of diverting mixture during the injecting operation. Specifically, Frenier states that, during the matrix acidizing process, "[p]ackers can

be used in the wellbore to control the formation zones into which the treatment fluid is injected from the wellbore." Ex. 1004 at 7:27–29. Moreover, Frenier incorporates by reference in its entirety U.S. Pat. No. 5,203,413 to Zerhboub, entitled "Product and Process for Acid Diversion in the Treatment of Subterranean Formations," issued April 20, 1993 ("Zerhboub") (Ex. 1014). Zerhboub claims the invention of "[a] process for treating a relatively low permeability first zone of a subterranean formation…." Ex. 1014 at 8:44–45. The claimed invention comprises the steps of: (a) injecting a foam … (b) injecting a treatment fluid … and (c) repeating said sequential foam and treatment fluid injecting steps (a) and (b). Ex. 1014 at 8:44–60. Zerhboub further clarifies that the injected foam of step (a) is a diverting mixture. Ex. 1014 at 4:1–5; 4:36–37 ("The principle of diversion by means of a foam is also known.).

Moreover, Reyes discloses the use of additives, such as "particulate diverters." Ex. 1005 at 11:34-54.

It would have been obvious to a POSITA in light of Frenier and Reyes to alternately inject a first amount of treatment mixture and a second amount of a diverting mixture into the well to better control the placement of the treatment fluid into the formation zones and avoid so-called "thief zones." Ex. 1003 at ¶ 299.

9. Dependent Claim 9

a. "The method of claim 1 wherein the injecting operation further comprises: injecting the treatment

> **mixture until the pressure within the well reaches a predetermined target pressure calculated based on the fracture pressure of the nano-darcy shale formation;"**

Frenier discloses injecting the treatment mixture until the pressure within the well reaches a predetermined target pressure calculated based on the fracture pressure of the nano-darcy shale formation. Notably, the '843 Patent provides that "the target pressure may be the fracture pressure for the formation…." Ex. 1001 at 8:65–66. Frenier states, "[o]ne of ordinary skill in the art will recognize that the well treatment compositions of the present invention can be used in the fracture acidizing of a formation. **By increasing the pumping pressure (to above the minimum in situ rock stress), a matrix acidizing treatment becomes an acid fracturing treatment**." *Id*. at 4:49–53 (emphasis added). Thus, Frenier describes injecting the treatment mixture until the pressure within the well reaches fracture pressure, i.e., "a predetermined target pressure calculated based on the fracture pressure of the nano-darcy shale formation." Ex. 1003 at ¶¶ 302–03.

Likewise, in a matrix acidizing method operation, the predetermined target pressure calculated based on the fracture pressure of the nano-darcy shale formation is simply any predetermined pressure less than that of the fracture pressure. Ex. 1004 at 4:49–53.

Reyes more clearly states, "[o]ne of ordinary skill in the art will understand how to measure the fracture gradient of a subterranean formation and **how to**

introduce a treatment fluid to a subterranean formation above or below this pressure." Ex. 1005 at 9:44–53 (emphasis added).

This claim element would have been obvious to a POSITA because it would have been desired to predetermine a pressure that is less than the fracture pressure but still sufficient to cause the treatment fluid to disperse into the subterranean formation and then inject the treatment mixture until the well reaches that predetermined pressure. Ex. 1003 at ¶ 306.

b. "and upon reaching the target pressure, shutting in the well."

Frenier explicitly discloses shutting in the well upon reaching the target pressure. Specifically, Frenier states, "[a]fter the composition has been injected into the formation, optionally the well can be shut in for a period of time to allow more complete reaction between the acid and the formation material." Ex. 1004 at 7:30–33.

This method would have also been obvious to POSITA, as stated by the Examiner, to "have more control over the contact time between the mixture and the well." Ex. 1002 at 252; Ex. 1003 at ¶ 308; Ex. Ex. 1014 at 7:25–29 ("Stage 3: Shut-in of the well. The decompression destabilizes the foam primarily in the low permeability cell. As indicated above, the advantage of a shut-in is that diversion is accelerated.").

It would have been obvious to a POSITA in light of Frenier and Reyes prior

to the effective filing date of the '843 Patent to shut in the well upon reaching the target pressure. Ex. 1003 at ¶ 309.

10. Dependent Claim 10

a. "The method of claim 1, further comprising: injecting the treatment mixture containing a metal complexing agent in combination with hydraulic fracturing and injecting into the formation in conjunction with propagation of induced fractures,"

Frenier states that "one can perform a stimulation technique by… injecting chemicals through the wellbore and into the formation at pressures sufficient to fracture the formation, thereby creating a large flow channel though [*sic*] which hydrocarbon can more readily move from the formation and into the wellbore." Ex. 1004 at 1:40–54.

However, Reyes explicitly discloses that treatment fluids may be used in conjunction with treatment operations such as "drilling operations, **stimulation operations, production operations, remediation operations**, sand control treatments, and the like." Ex. 1005 at 1:13–17 (emphasis added). Thus, it would have been obvious to a POSITA in light of Frenier and Reyes prior to the effective filing date of the '843 Patent to "inject[] the treatment mixture containing a metal complexing agent in combination with hydraulic fracturing and injecting into the formation in conjunction with propagation of induced fractures." Ex. 1003 at ¶ 313.

b. "the metal complexing agent including citric acid or EDTA."

Petitioner incorporates its arguments above with respect to dependent claims 2, 3, and 5. Both Frenier and Reyes teach selecting either citric acid or EDTA to use as the metal complexing agent. Thus, such would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier alone or in combination with Reyes. Ex. 1003 at ¶ 315.

11. Dependent Claim 11

a. "The method of claim 1 further comprising: monitoring at least one of pH and well pressure during the maintaining operation;"

Frenier explicitly discloses the use of "pressure transient analysis" during the maintaining operation: "[t]he efficiency of a matrix treatment depends primarily upon removing or bypassing regions of low permeability that restrict productivity…. **Pressure transient analysis** is a common technique for estimating the extent of damage." Ex. 1004 at 6:49–64 (emphasis added). As a POSITA would have known and understood prior to April 14, 2014, pressure transient analysis includes monitoring well pressure over time while the matrix acidizing operation is taking place. Ex. 1003 at ¶¶ 316–17.

Additionally, a POSITA would have known and understood, or at least it would have been obvious, to monitor at least one of the pH and well pressure during the maintaining operation to determine when the well stimulation operation had either removed the blockage in the well or the reactants in the injected fluids had

been spent. Ex. 1003 at ¶ 318. Monitoring at least one of the pH and well pressure while the treatment mixture contacts the subterranean formation has been common practice in the industry since well-before the effective filing date of the '843 Patent. Ex. 1003 at ¶ 320.

b. "and initiating the removing operation based on results of the monitoring operation."

As mentioned above, Frenier discloses the use of "pressure transient analysis" during the maintaining operation: "[p]ressure transient analysis is a common technique for estimating the extent of damage." Ex. 1004 at 6:49–64. It would have been obvious to a POSITA before the effective date of the '843 Patent to remove the treatment mixture from the well after the contact time based on the results of the pressure transient analysis. Ex. 1003 at ¶ 321–322.

Specifically, it would have been obvious to a POSITA, to perform this step so as to remove the metals from the formation before they begin to precipitate and congest the well. Ex. 1003 at ¶ 323. Frenier discusses the need for avoiding precipitation after contact with the formation. Ex. 1004 at 3:23–47; 8:12–16.

12. Dependent Claim 12

a. "The method of claim 1 further wherein the metal complexing agent is selected from glutamic acid diacetic acid (GLDA), methylglycine diacetic acid (MGDA), β-alanine diacetic acid (β-ADA), ethylenediaminedisuccinic acid, S,S-ethylenediaminedi succinic acid (EDDS), iminodisuccinic acid (IDS), hydroxyiminodisuccinic

> **acid (HIDS), polyamino disuccinic acids, N-bis[2-(1,2-dicarboxyethoxy)ethyl]glycine (BCA6), N-bis[2-(1,2-dicarboxyethoxy)ethyl]aspartic acid (BCA5), N-bis[2-(1,2-dicarboxyethoxy)ethyl]methylglycine (MCBA5), N-tris[(1,2-dicarboxyethoxy)ethyl]amine (TCA6), N-methyliminodiacetic acid (MIDA), iminodiacetic acid (IDA), N-(2-acetamido)iminodiacetic acid (ADA), hydroxymethyl-iminodiacetic acid, 2-(2-carboxyethylamino) succinic acid (CEAA), 2-(2-carboxymethylamino) succinic acid (CMAA), diethylenetriamine-N,N″-disuccinic acid, triethylenetetramine-N,N‴-disuccinic acid, 1,6-hexamethylenediamine-N,N′-disuccinic acid, tetraethylenepentamine-N,N⁗-disuccinic acid, 2-hydroxypropylene-1,3-diamine-N,N′-disuccinic acid, 1,2-propylenediamine-N,N′-disuccinic acid, 1,3-propylenediamine-N,N′-disuccinic acid, cis-cyclohexanediamine-N,N′-disuccinic acid, trans-cyclohexanediamine-N,N′-disuccinic acid, ethylenebis(oxyethylenenitrilo)-N,N′-disuccinic acid, glucoheptanoic acid, cysteic acid-N,N-diacetic acid, cysteic acid-N-monoacetic acid, alanine-N-monoacetic acid, N-(3-hydroxysuccinyl) aspartic acid, N-[2-(3-hydroxysuccinyl)]-L-serine, aspartic acid-N,N-diacetic acid, aspartic acid-N-monoacetic acid, any salt thereof, any derivative thereof, or any combination thereof."**

Frenier discloses the metal complexing agent selected from several, if not all, of the acids listed in claim 12. Frenier discloses the use of organic acids as complexing agents. Ex. 1004 st 5:16–20. A POSITA would have known and understood Frenier's disclosure of organic acids as metal complexing agents to include the listed biodegradable acids of claim 12. Ex. 1003 at ¶¶ 326–27.

Reyes provides a nearly identical list of biodegradable chelating agents to the

list of claim 12. Ex. 1005 at 10:24–58 (not reproduced here for brevity). Thus, Reyes discloses the metal complexing agent selected from several, if not all, of the acids listed in claim 12. It would have been obvious to a POSITA to select any of the biodegradable chelating agents listed in claim 12 as the metal complexing agent. Ex. 1003 at ¶ 329.

13. Dependent Claim 13

a. "The method of claim 1 further wherein the metal complexing agent is selected from acetic acid, acrylates, dihydroxymaleic acid, salts of dihydroxymaleic acid, EDTA (ethylenediamine tetraacetic acid), salts of EDTA, erythorbic acid, erythroboric acid, formic acid, gluconodeltalactone, GLDA (glutamic acid N,N-diacetic acid), salts of GLDA, HEDTA (hydroxyethylenediamine triacetic acid), salts of HEDTA, HEIDA (disodium ethanoldiglycine), salts of HEIDA, MGDA (methylglycine N,N-diacetic acid), salts of MGDA, NTA (nitriolotriacetic acid), organic metal complexers, ligands, porphyrins, pigments, peptides, saccharides, nucleic acids, phosphonic acid, polyacrylic acid and citric acid in an amount sufficient to sequester at least a portion of a metal compound."

As set forth above, Frenier discloses selecting HEDTA or HEIDA as the metal complexing agent. Ex. 1004 at 4:13–17

Reyes discloses several, if not all, of the acids listed in claim 13. Ex. 1005 at 10:59–67; 11:1–4 (not reproduced herein for brevity).

It would have been obvious to a POSITA in light of Frenier alone or with

Reyes to select any of the chelating agents listed in claim 13 as the metal complexing agent "in an amount sufficient to sequester at least a portion of a metal compound." Ex. 1003 at ¶¶ 332–33.

VIII. CONCLUSION

Petitioner respectfully submits there is a reasonable likelihood it will prevail in establishing that the Challenged Claims are unpatentable and requests that trial be instituted.

Respectfully submitted,

Date: April 17, 2024

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CERTIFICATION OF WORD COUNT

This Petition complies with the type-volume limitations as mandated in 37 C.F.R. § 42.24, totaling 13,964 words. Counsel has relied upon the word count feature provided by Microsoft Word.

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CERTIFICATE OF SERVICE

I hereby certify pursuant to 37 C.F.R. §§ 42.6(e) and 42.105 that on this 17th day of April 2024, a true and correct copy of the foregoing Petition for *Inter Partes* Review of U.S. Patent 9,944,843 was served via Federal Express upon the following counsel of record for Patent Owner Flex-Chem Holding Company, Inc.:

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UNITED STATES

I further hereby certify that on this 17th day of April, 2024, a true and correct copy of the foregoing Petition for Inter Partes Review of U.S. Patent 9,944,843 was served via email upon the following litigation counsel for Patent Owner Flex-Chem Holding Company, Inc.:

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Date: April 17, 2024

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